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DESIGN AND DEVELOPMENT OF A HAZARDOUS WASTE REACTIVITY TESTING PROTOCOL

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16. ABSTRACT

A test protocol to determine the gross chemical composition of waste materials has been developed for use at uncontrolled hazardous waste sites. Included is a field test kit, flow diagrams, a descriptive manual and a mixing device to observe the effects of mixing two hazardous wastes. Prior knowledge of the chemical composition of the waste materials is not required for this protocol.

Unidentified wastes were classified by reactivity groups using this test scheme in the laboratory and in the field. Small amounts of wastes that indicated compatibility were placed in the mixing device to verify that mixing would not generate immediate catastrophic results.

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FOREWORD

The U.S. Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimonies to the deterioration of our natural environment. The complexity of that environment and the interplay of its components require a concentrated and integrated attack on the problem.

Research and development is that necessary first step in problem solution, and it involves defining the problem, measuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems to prevent, treat, and manage wastewater and solid and hazardous waste pollutant discharges from municipal and community sources, to preserve and treat public drinking water supplies, and to minimize the adverse economic, social, health, and aesthetic effects of pollution. This publication is one of the products of that research and is a most vital communications link between the researcher and the user community.

Waste materials must often be recontainerized during cleanup of uncontrolled or abandoned hazardous waste dump sites under the activities funded by regulations to the Resource Conservation and Recovery Act (Superfund). The potential for catastrophic reactions when unknown materials are mixed is high. To lessen the degree of risk we have prepared a hazardous waste reactivity test protocol by which unknown materials may be classified into various reactivity categories. This report documents the preliminary development of the protocol and the test kit used to conduct the protocol in the field.

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ABSTRACT

A project was conducted to develop a test scheme (protocol) to determine the gross chemical composition of waste materials in the field. Such a test scheme is needed during remedial actions at hazardous waste disposal sites, where it is necessary to predict the potential consequences of mixing wastes from separate sources. Earlier procedures have assumed a prior knowledge of the chemical composition of the wastes. Information obtained from these tests is used to classify wastes into reactivity groups and thus predict compatibility.

The test scheme developed here includes a field test kit, a series of flow diagrams, and a manual for using the flow diagrams and test procedures. Because small-scale mixing is needed as a safeguard before large-scale mixing takes place (even when the chemical composition of two wastes indicates compatibility), a simple device is included for observing the effects of mixing two hazardous materials.

The protocol was challenged with more than 60 compounds in the laboratory and 28 waste samples in the field. Of 755 laboratory observations, 15 were false positives and 2 were false negatives (including replicate tests). All but one of the field samples were classified into the correct reactivity group based on the bulk chemical composition listed in the suppliers' manifest. The one incorrectly identified sample was found to be incorrectly labeled by the supplier and was correctly classified according to its actual composition.

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A special acknowledgment is also due to the Project Officer, Ms. Naomi Barkley. Her insight into the necessity of transforming this work from a technical exercise into a useful tool, and her continued enthusiasm in helping us overcome often convoluted problems of chemical logic were also crucial to the success of this project.

SECTION 1

INTRODUCTION

During remedial action at an abandoned waste disposal site, it is frequently necessary to determine whether waste materials from separate sources can be safely mixed for bulk recontainerization, shipment, and ultimate proper disposal. At operating Resource Conservation and Recovery Act (RCRA) facilities, waste composition may need to be verified against shipper manifests. The history of hazardous waste management contains numerous accounts of disasters resulting from the mixing of incompatible wastes. Proper management of hazardous waste therefore requires test procedures to identify potential reactivity hazards and avoid the consequences of mixing such hazardous wastes.

A document has been developed that classifies wastes by chemical class or general reactive properties or both and lays out a sequence of activities to determine the compatibility of two wastes (Reference 1). The key elements of this document are a compatibility chart and a flow diagram for its use. But, the authors presuppose a knowledge of the waste composition. This information is often available from generators of hazardous wastes, but during remedial action at an abandoned hazardous waste disposal site, it is seldom available. The need to test mix two wastes on a small scale is emphasized even if the compatibility chart indicates compatibility.

The purpose of the present study is to establish a series of qualitative test procedures that will enable field workers to classify hazardous waste materials according to their gross chemical composition when little or no prior knowledge is available regarding their components. Information about chemical composition is then used to predict which waste materials can be mixed safely before actually performing mixing tests. Note that only qualitative results are obtained, and thus for low-level contaminants, overly conservative conclusions could be drawn.

Because of the environment in which these classification test procedures will be used, limitations on the schemes, test procedures, and equipment have been addressed during the course of this study. These limitations are:

- The procedures must be safe and easy to perform by nonchemists
- The procedures must give definitive, objective results

- The equipment must be standard, readily available, portable, and use a minimal number of utility hookups, since the tests may be applied in remote field locations

- The procedures must be performed as rapidly as possible, since numerous waste samples may require testing

- The test procedures must be able to be performed with slightly restricted manual dexterity, since they may be performed in areas where protective clothing is required

Standard organic and inorganic qualitative test procedures have been identified from the literature. These test procedures have been organized into a scheme for classification of waste materials according to their chemical composition. These procedures have been evaluated by applying them to a set of 58 reference compounds chosen to represent materials likely to be found at hazardous waste disposal sites.

A field test kit has been assembled that contains all necessary equipment and materials for conducting the test procedures. A device for determining the effects of mixing hazardous wastes has also been assembled and is included in the field test kit. The mixing device enables investigators to observe gas and heat evolved when small quantities of waste materials are mixed. The test kit includes a complete manual for its use (Appendix A).

The test kit has been used in a week-long field evaluation at the EPA Combustion Research Facility located in Jefferson, Arkansas. During the field evaluation, the test procedures were applied to 25 actual waste samples. The protocol has been shown to be applicable to mixtures of materials.

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

A series of qualitative test procedures has been incorporated into test schemes for categorizing hazardous waste materials. Test results are used to classify waste materials according to their gross chemical composition. All reagents and equipment required for the tests have been incorporated into a field test kit, which includes a manual for its use.

The test schemes have been verified using representative pure compounds and mixtures of compounds. Actual results have been compared with the results that were expected because of chemical structures of the reference compounds. Each test scheme was shown to provide reliable results for the reference compounds. The schemes were applied to the reference compounds in blind tests by two different analysts to verify the procedure sequences. During initial verification, more than 750 individual observations were made, with 15 false positive and 2 false negative observations recorded.

The test kit was used in a field evaluation at the EPA Combustion Research Facility in Jefferson, Arkansas. During the field evaluation, 25 hazardous waste materials with a total of 29 phases were examined using the test procedures. The test results provided accurate descriptions of all of the phases based on haulers' records for the waste materials. An average of less than 90 min was required for the testing of a single waste material. The tests were performed under simulated field conditions.

The field evaluation of the test kit demonstrates three important positive aspects of the test kit:

- The test procedures were shown to be applicable to field conditions
- The test procedures provided accurate descriptions of actual hazardous wastes
- The complete categorization of an unknown waste required an average of less than 90 min

In general, the kit and protocol meet all the criteria listed in Section 1. The tests are simple and can be conducted by nonchemists with some training. However, some laboratory experience is highly desirable. The results of the procedures are definitive in almost every case. For those not definitive, an hour or two of training is adequate for the technician to be able to correctly interpret the results. All equipment except specially constructed shipping boxes and the glass reactor vessel for the mixing device is standard. The time requirements, although not as short as could be desired, are brief when compared to normal laboratory analysis.

Significant weaknesses were the lack of identification of trace materials in complex matrices and the identification of water-based organics. The field use of the kit may also be limited by weather conditions. The kit probably cannot be used in conditions where the temperature is less than 5°C (40°F) or greater than 33°C (90°F), when wind is greater than 15 mph, or during precipitation (i.e., rain, snow, etc.).

Based on the results of the laboratory development and particularly on the results of the field evaluation, the test kit has considerable potential for applications in hazardous waste management. This approach presents an attractive alternative to laboratory analysis for the qualitative gross categorization of waste materials.

Though the primary purpose of the development effort was to determine compatibility characteristics, the test results can potentially be used to:

- Predict best disposal technology
- Identify generic wastes
- Predict degree of hazard

Additional testing of real waste materials should be undertaken to demonstrate further the applicability of the test kit to actual hazardous waste materials.

The field testing of additional waste materials from a wide range of chemical classes should identify any remaining limitations of the test procedures or the test kit and provide a statistical basis for their general acceptance.

Additional development effort is needed to identify specific tests for three reactivity groups for which test procedures have not been found -- epoxides, isocyanates, and nitrides. In addition, general tests are needed for polymerizable materials and organics dissolved in water, and a more definitive means for identifying explosives is required.

Finally, the test procedures should be challenged from the viewpoint of detection limits. All procedures are currently expected to give positive results at the 20-percent level, and they may give positive results at the 5-percent level. These detection limits must be verified and documented.

SECTION 3

DISCUSSION

A series of test procedures (References 2, 3, 4, and 5) has been assembled to classify hazardous wastes based on their gross chemical composition. These test procedures are based on established qualitative chemical tests. Specific functional group tests have been identified for all but 5 of the 41 reactivity groups (RGN's) listed in Reference 1. A complete listing of tests by reactivity group is presented in Table 1. Details of the test procedures are lengthy and are therefore described fully in the field test kit manual (Appendix A).

Basically five types of tests are involved in the classification sequence:

- Physical observation
- Treated paper tests
- Ignition tests
- Solubility-reactivity tests
- Functionality tests

The latter two are spot-plate or small-test-tube tests.

Physical examination of the waste material reveals the phase (liquid, sludge, solid) and may give clues about the material present (elemental metals, for example). Separate test paper is used to conduct tests for pH, and for oxidative and reductive potential. Ignition (flame) tests are performed using a small crystal or droplet of material first on a nichrome wire then on a spatula or in a small crucible. Combustible materials are classified as organics, and explosives are identified. Solubility-reactivity tests are performed by treating small amounts of the waste material with a few drops each of various acids and solvents. Functionality tests for sulfide and cyanide are performed concurrently. Additional functionality tests are used to make a final assignment of waste materials into reactivity groups based on the functional group present. These tests are based on

TABLE 1. SUMMARY OF SPECIFIC TEST PROCEDURES

RGN	Reactivity group name	Name of test	Reference
1	Acids, mineral, nonoxidizing	pH	Paper test
2	Acids, mineral, oxidizing	pH, oxidation	Paper tests
3	Acids organic	pH, flame	Paper test
4	Alcohols and glycols	Vanadate	4, p. 176
5	Aldehydes	Dinitrophenylhydrazine, Schiff's test	2, pp. 264-65
6	Amides	Oxamide test	4, pp. 264-65
7	Amines, aliphatic and aromatic	p-Nitrobenzenediazonium-tetrafluoroborate	4, p. 240
8	Azocompounds, diazo compounds, and hydrazines	Dimethylaminobenzaldehyde, glutaconic aldehyde	4, p. 666 4, p. 269
9	Carbamates	Diphenylcarbohydrazide	4, p. 390
10	Caustics	pH	Paper test
11	Cyanides	Ferrous sulfate, ferric chloride	2, pp. 172, 174
12	Dithiocarbamates	Cupric chloride, acetic acid	4, pp. 304-05
13	Esters	Hydroxamate	4, p. 214
14	Ethers	Iodine test	2, p. 267
15	Fluorides, inorganic	Zirconium alizarinate	4, p. 420
16	Hydrocarbons, aromatic	Friedel-Crafts and ferrox test	2, pp. 233-34 2, pp. 231-32
17	Halogenated organics	Sodium fusion	2, pp. 173, 177-80
18	Isocyanates	No test identified	--

(continued)

TABLE 1. (continued)

RGN	Reactivity group name	Name of test	Reference
19	Ketones	Dinitrophenylhydrazine, Schiff's test	2, pp. 264-65
20	Mercaptans and other organic sulfides	Lead acetate	2, p. 274
21	Metals, alkali and alkaline earth, elemental and alloys	Water, hydrochloric acid, phosphomolybdic acid	3, p. 362
22	Metals, other elemental in the form of powders, vapors, or sponges	Phosphomolybdic acid	3, p. 362
23	Metals, other elemental, and alloys as sheets, rods, moldings, drops, etc.	Phosphomolybdic acid	3, p. 362
24	Metals and metal compounds, toxic	Zinc sulfide, ammonium sulfide	5, pp. 134-35
25	Nitrides	No test identified	--
26	Nitriles	Oxamide test	4, pp. 264-65
27	Nitro compounds	Tetrabase fusion	4, p. 295
28	Hydrocarbon, aliphatic, unsaturated	Baeyer test, ferrox test	2, p. 162 2, pp. 231-32
29	Hydrocarbon, aliphatic, saturated	Ferrox test	2, pp. 231-32
30	Peroxides and hydroperoxides, organic	Potassium iodide-starch	4, p. 615
31	Phenols and creosols	Ferric chloride test	2, p. 228
32	Organophosphates, phosphothioates, and phosphodithioates	Sodium fusion	2, p. 173 ff

(continued)

TABLE 1. (concluded)

RGN	Reactivity group name	Name of test	Reference
33	Sulfides, inorganic	Lead acetate	Paper test
34	Epoxides	No test identified	--
101	Combustible and flammable materials, miscellaneous	Flame test	2, pp. 160-61
102	Explosives	Flame test	2, pp. 160-61
103	Polymerizable compounds	No test identified	--
104	Oxidizing agents, strong	Iodide-starch	Paper test
105	Reducing agents, strong	Methylene blue	Paper test
106	Water and mixtures containing water	Copper sulfate	2, p. 161
107	Water-reactive substances	Water spot test	Spot test
	Other Test Procedures		
Organic	All RGN's	Ferrox test	2, pp. 231-32
Organic	All RGN's with heteroatoms (N, O, P, S, X)	Sodium fusion	2, p. 172 ff
All	All RGN's	Flame test	2, pp. 160-1

established qualitative organic and inorganic procedures and are referenced in Table 1.

WARNING

> The detection limits of these tests in some cases may not be sufficient to identify trace quantities of materials (e.g., cyanide at several parts per million) that could be hazardous to personnel if subjected to acid treatment.

3.1 ORGANIZATION OF TEST PROCEDURES

The procedures have been organized into a visual examination phase and six procedure sets (PS), as outlined below:

- PS 1 -- pH and redox test to identify acids, caustics, oxidizing and reducing agents
- PS 2 -- Solution-reactivity and special functionality tests to determine reactivity and/or solubility in a series of acids and solvents and identify the presence of water, sulfide, and cyanide
- PS 3 -- Flame tests to identify combustible (organic) and extremely heat-sensitive (explosive) materials (additional "indicators" of composition may also be obtained)
- PS 4 -- Ferrox and sodium fusion tests to provide elemental analysis for organic waste materials
- PS 5 -- Functional group tests for specific identification of organic functional groups (RGN's)
- PS 6 -- Functional group tests for specific identification of inorganic RGN's

The procedure sets have been organized into a master scheme for examination of unknown waste materials. The organizational sequence of procedure sets into a general scheme is described in the following paragraphs.

Test procedures are organized into a scheme for the classification of unknown hazardous wastes into reactivity groups (References 2, 3, 4, and 5). Preliminary procedures classify the material as acid, base, oxidizing, reducing, or water reactive, and primarily organic or inorganic. The results of preliminary tests are used to give direction to subsequent testing procedures which are used for further classification, ultimately into reactivity groups. The scheme is organized in a manner such that materials with high reactivity or unusual hazards are identified early in the testing sequence. In some cases, information gained in the early stages of testing can be used to classify the material and eliminate the need for further testing, provide cautions to be applied in subsequent testing activities, or indicate the need for direction by qualified supervisory personnel.

The sequence in which test procedures are carried out is shown schematically in Figure 1. The testing sequence begins with a visual examination of the waste material to classify it as a solid, sludge, slurry, or liquid. This examination may give a strong indication of its identity (i.e., metal).

The testing for all four classes is initiated with PS 1 (pH and redox tests). This procedure is followed because the most extreme hazards of liquids -- strong acidity and/or strongly oxidizing properties -- can be detected by tests in PS 1 and the most extreme hazards anticipated from solids is their explosiveness which can be detected by the flame test (PS 3). Acidity and oxidative potential is a secondary hazard for solids and explosive solids are not expected to cause a problem in the PS 1 test sequence. If the material is strongly acidic, strongly oxidizing, or caustic, it is classified into the proper reactivity group; RGN 1, 3, 2, or 10, respectively. Further testing can be eliminated since these reactivity classes are incompatible with most other classes of wastes.

Slurries are separated by filtration into solid and liquid portions, if possible, after the pH and redox tests have been conducted and testing is continued separately for each of the phases. Likewise, two-phase liquids are separated and tests are conducted on the separate phases.

The next test set is PS 2 (for both liquids and solids) to determine the solubility and reactivity properties of the waste material with acids and organic solvents. The flame test (PS 3) is carried out to determine flammability and explosiveness. Results from PS 2 and PS 3 help identify whether the waste material is primarily organic or inorganic, and whether it has the potential to form extremely hazardous compounds when mixed with water or acids (hydrogen cyanide, hydrogen sulfide).

For organic materials, functionality tests (PS 4 and PS 5) are performed to assign specific RGN's to the waste material. Inorganic materials are classified into specific RGN's using PS 6.

3.2 REFERENCE COMPOUNDS

A series of 58 compounds was selected from those listed in Appendix 1 of Reference 1. These compounds, which represent materials likely to be found in hazardous waste sites, were selected using the following criteria:

- Compounds had to be commercially available
- At least one compound was selected for each reactivity group

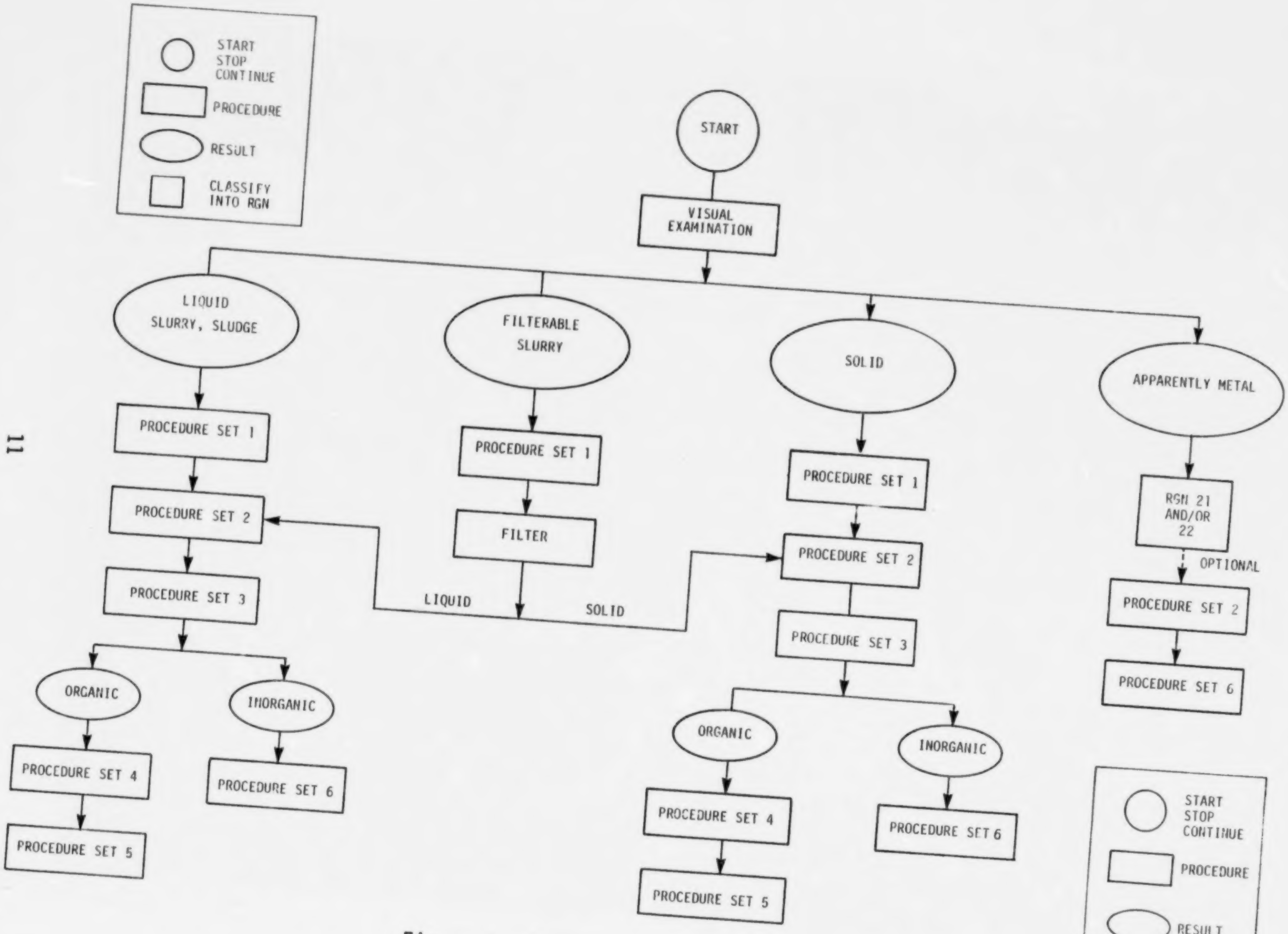

START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY
INTO RGN
START
VISUAL
EXAMINATION
LIQUID
SLURRY, SLUDGE
FILTERABLE
SLURRY
SOLID
APPARENTLY METAL
PROCEDURE SET 1
PROCEDURE SET 2
PROCEDURE SET 3
ORGANIC
INORGANIC
PROCEDURE SET 4
PROCEDURE SET 5
PROCEDURE SET 6
PROCEDURE SET 1
FILTER
LIQUID
SOLID
PROCEDURE SET 1
PROCEDURE SET 2
PROCEDURE SET 3
ORGANIC
INORGANIC
PROCEDURE SET 4
PROCEDURE SET 5
PROCEDURE SET 6
RGN 21
AND/OR
22
OPTIONAL
PROCEDURE SET 2
PROCEDURE SET 6
START
STOP
CONTINUE
PROCEDURE
RESULT

Figure 1. Sequence of procedure sets.

- Polyfunctional materials were selected when possible, so that reactivity groups would be well represented by the reference compounds, and so that potential interferences could be introduced to simulate mixtures likely to be found in real waste samples

A list of the reference compounds chosen for this study and the assigned reactivity groups for each compound is presented in Table 2. All reactivity groups are represented except RGN 25 (nitrides). These materials are not commercially available and are extremely dangerous to synthesize, so they were omitted from the investigation.

3.3 DESCRIPTION OF PROCEDURE SETS

Procedures employed for the examination of hazardous waste materials are broken into six procedure sets which were briefly outlined in Section 3.1. Results obtained early in the testing sequence are used to give direction to subsequent testing procedures. The organization of procedure sets into an overall testing sequence is described in Section 3.1. Detailed descriptions of the six procedure sets are presented in the following paragraphs. The test procedures were verified using the reference compounds (Section 3.2). The first step in testing is determination of pH and redox potential.

3.3.1 PS 1 -- pH and Redox Tests

Oxidation, reduction, and pH tests are performed on the waste material using appropriate test papers. Some of the most reactive waste materials (as judged by incompatibilities according to the compatibility chart) are determined by these simple tests, so these are performed early in the testing sequence.

Determination of pH is made by means of pH indicator strips (Merck). Mineral acids (RGN 1) are arbitrarily designated as those materials having a pH of less than 1.5, and materials are classified as organic acids (RGN 3) if the pH is 1.5 to 4.5. Definitive distinction between these two classes is made by means of one flame test (PS 3), but for purposes of determining compatibility characteristics the delineation of these two classes by the simple pH test will suffice. Materials are classified as caustic (RGN 10) if they have a pH of 10.5 or greater.

Oxidizing agents (RGN 104) are determined with (commercially available) starch-iodide paper, and reducing agents (RGN 105) are detected using (laboratory prepared) methylene blue paper. Oxidizing acids (RGN 2) are those materials with a pH less than 2 to 3 with a positive oxidation test.

For the purposes of predicting compatibility characteristics, classification of a waste material as a strong acid (RGN 1 or 2) or as a strong oxidizing agent (RGN 104) adequately defines the waste material such that no further testing is necessary. This is because acids and oxidizing agents are listed as incompatible by the compatibility chart with most other classes of waste materials. Similarly, if the material has the combined

TABLE 2. REFERENCE COMPOUNDS FOR HAZARDOUS WASTE COMPATIBILITY TESTING

	Reactivity Group Number (RGN)																																								
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	101	102	103	104	105	106	107
Acrolein					X																																X				
Aldicarb									X																																
N-Butyl acrylate													X																												
Diacetone alcohol				X															X																						
Ethyl acrylate													X																								X				
Fluoroboric acid	X														X																										
Hexafluorophosphoric acid	X														X																										
Potassium cyanide											X																														
Hydrofluoric acid	X														X																										
Hydroxyacetophenone																			X												X										
Lannate									X																																
Mercaptobenzothiazole																				X																					
Mercaptoethanol				X																X																					
Methyl methacrylate													X																								X				
Peracetic acid			X																											X											
Polypropylene																													X						X						
Polysulfide polymer														X						X															X				X		
Propylene oxide																																		X			X				
Vinyl acetate													X																								X				
Selenium diethyldithiocarbamate												X												X																	
TEPA																																X									
Ziram												X												X																	
Arsenic pentasulfide																								X									X								
Cadmium																							X	X																	
Chromium																							X	X																	
Copper																							X	X																	
Lead																							X	X																	
Nickel																						X		X																	
Trinitro benzoic acid			X																								X									X					
Ethylene glycol monomethyl ether				X										X																											
Styrene																X												X								X					
Chlorophenyl isocyanate																	X	X																							X
Bromoxynil																	X									X					X										
Chromic anhydride		X																						X														X			
Dipicryl amine							X																				X									X					
Barium																					X			X																	X
Sodium																					X																		X		X
Barium iodide																								X																	
Barium oxide										X														X																	X
Picric acid																											X				X					X					
Acetamide						X																																			
Aminopropionitrile							X																			X															
Aminothiazole							X																																		
Benzoyl peroxide																														X						X					
Calcium hypochlorite										X																												X			
Decane																													X												
1-decene																												X													
Dimethylaminoazobenzene							X	X																																	
Diphenamide						X																																			
Hexane																													X												
1-hexene																												X													
Hydroxylamine hydrochloride																																							X		
Malathion																																X									
Malonic nitrile			X																							X															
Naphthalene																X																									
Parathion																																X									
Toluene diisocyanate																		X																							
Totals by class	3	1	3	3	1	2	5	1	2	2	1	2	4	2	3	2	2	2	2	3	2	1	4	12	0	3	3	3	3	2	3	3	1	1	2	5	5	2	3	0	4

properties of caustic (RGN 10) and reducing agent (RGN 105), further testing is probably unnecessary for the purpose of compatibility prediction. It is recommended, however, that PS 3 be conducted on those materials falling into RGN's 1, 2, 104, or 10 and 105 so that explosive hazards do not go undetected.

A flow diagram for PS 1 is shown in Figure 2.

3.3.2 PS 2 -- Solution-Reactivity and Special Functionality Tests

Early in the testing sequence, the reactivity of waste material with water, mineral acids and bases, and common organic solvents is determined by direct treatment of the waste material with a series of reagents. The waste material is treated with water, nitric acid, hydrochloric acid, sulfuric acid, methanol, acetone, hexane, and toluene. The results of these drop tests give clues as to the identity of the waste material and also flag important potentials for reactions with materials likely to be found in hazardous waste disposal sites. If the material is found to be water reactive, (RGN 107) it is so labeled and further testing is suspended. (According to the compatibility chart, water-reactive materials should not be mixed with any other type of waste.)

A flow diagram for PS 2 is shown in Figure 3. Materials are first classified according to their reactivity or solubility in water. Materials are then tested for sulfide (RGN 33) and cyanide (RGN 11). If the material is found to contain sulfide or cyanide, further testing is suspended due to the hazard of handling these materials. Liquids are tested for the presence of water (RGN 106). Aqueous solutions are considered to be primarily inorganic (for the purposes of compatibility) and nonaqueous liquids are assumed to be organic.

Drop tests (the testing of the material with a few drops of selected solvents) are performed and materials may be tentatively classified as organic or inorganic, based upon their solubility characteristics. Generally, materials which are soluble in organic solvents are classified as organic. Confirmation of the organic/inorganic classification is made by means of the flame test (PS 3), discussed in the following section.

3.3.3 PS 3 -- Flame Test

Classification of a material as organic or inorganic is made by means of the flame test. Materials of unusual flammability (e.g., explosives -- RGN 102) are also identified in this manner. The test consists of placing a small portion of waste material on a nichrome wire in a propane flame. If no visible observation can be made with the nichrome wire test, then a slightly larger portion in a crucible spoon is subjected to the flame. The flow scheme for PS 3 is shown in Figure 4.

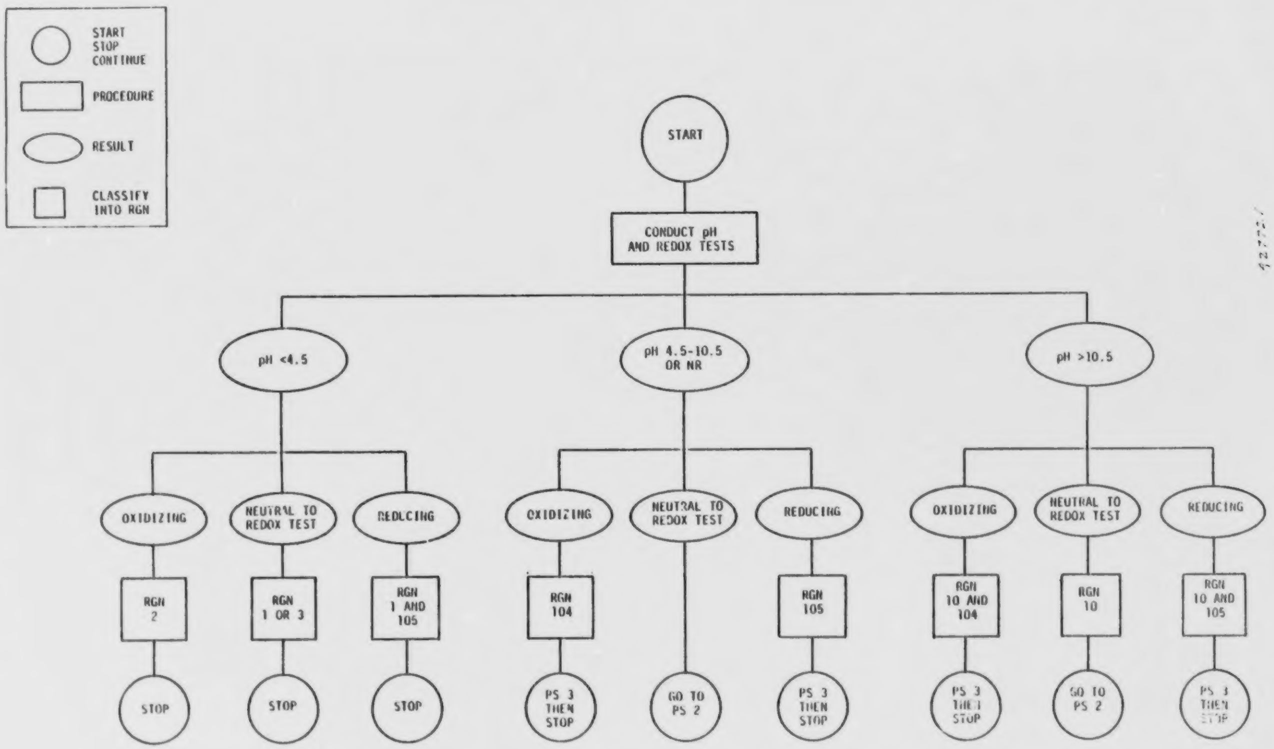

START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY
INTO RGN
START
CONDUCT pH
AND REDOX TESTS
pH <4.5
pH 4.5-10.5
OR NR
pH >10.5
OXIDIZING
NEUTRAL TO
REDOX TEST
REDUCING
OXIDIZING
NEUTRAL TO
REDOX TEST
REDUCING
OXIDIZING
NEUTRAL TO
REDOX TEST
REDUCING
RGN
2
RGN
1 OR 3
RGN
1 AND
105
RGN
104
RGN
105
RGN
10 AND
104
RGN
10
RGN
10 AND
105
STOP
STOP
STOP
PS 3
THEN
STOP
GO TO
PS 2
PS 3
THEN
STOP
PS 3
THEN
STOP
GO TO
PS 2
PS 3
THEN
STOP

Figure 2. Procedure set 1 -- pH and redox tests.

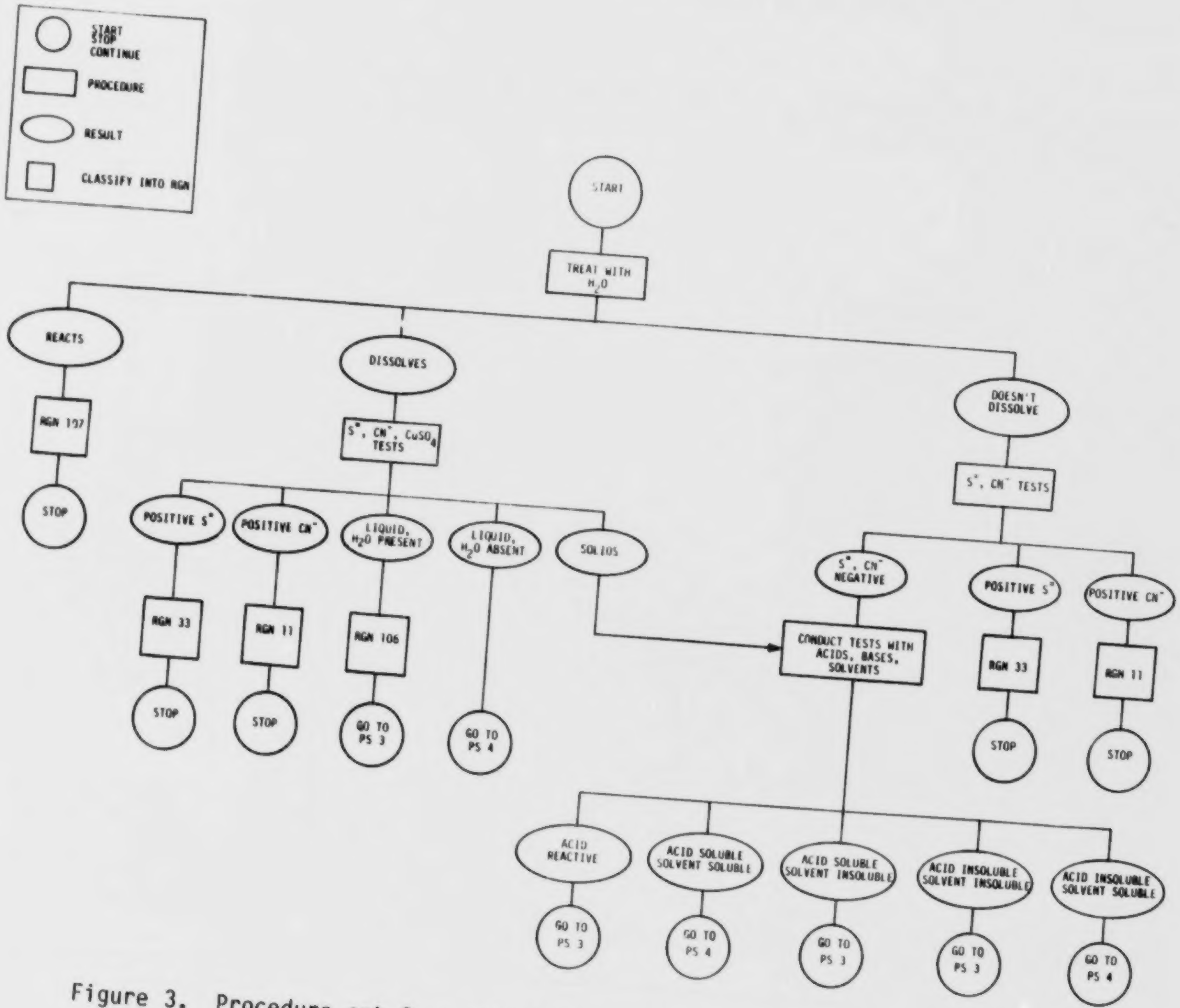

START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY INTO RGN
START
TREAT WITH H$_2$O
REACTS
RGN 107
STOP
DISSOLVES
S$^=$, CN$^-$, CuSO$_4$ TESTS
POSITIVE S$^=$
RGN 33
STOP
POSITIVE CN$^-$
RGN 11
STOP
LIQUID, H$_2$O PRESENT
RGN 106
GO TO PS 3
LIQUID, H$_2$O ABSENT
GO TO PS 4
SOLIDS
DOESN'T DISSOLVE
S$^=$, CN$^-$ TESTS
S$^=$, CN$^-$ NEGATIVE
CONDUCT TESTS WITH ACIDS, BASES, SOLVENTS
POSITIVE S$^=$
RGN 33
STOP
POSITIVE CN$^-$
RGN 11
STOP
ACID REACTIVE
GO TO PS 3
ACID SOLUBLE SOLVENT SOLUBLE
GO TO PS 4
ACID SOLUBLE SOLVENT INSOLUBLE
GO TO PS 3
ACID INSOLUBLE SOLVENT INSOLUBLE
GO TO PS 3
ACID INSOLUBLE SOLVENT SOLUBLE
GO TO PS 4

Figure 3. Procedure set 2 -- solution/reactivity and special functionality tests.

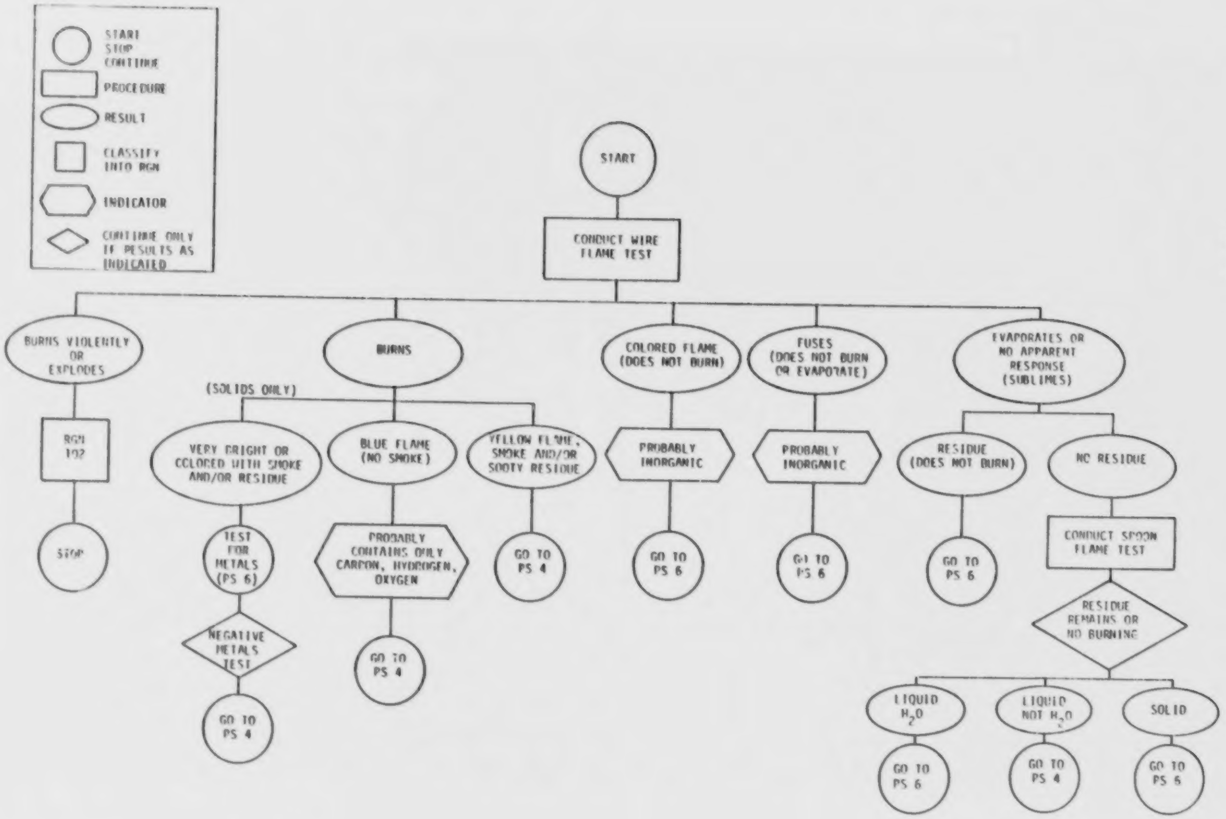

START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY INTO RGN
INDICATOR
CONTINUE ONLY IF RESULTS AS INDICATED
START
CONDUCT WIRE FLAME TEST
BURNS VIOLENTLY OR EXPLODES
RGN 102
STOP
BURNS
(SOLIDS ONLY)
VERY BRIGHT OR COLORED WITH SMOKE AND/OR RESIDUE
TEST FOR METALS (PS 6)
NEGATIVE METALS TEST
GO TO PS 4
BLUE FLAME (NO SMOKE)
PROBABLY CONTAINS ONLY CARBON, HYDROGEN, OXYGEN
GO TO PS 4
YELLOW FLAME, SMOKE AND/OR SOOTY RESIDUE
GO TO PS 4
COLORED FLAME (DOES NOT BURN)
PROBABLY INORGANIC
GO TO PS 6
FUSES (DOES NOT BURN OR EVAPORATE)
PROBABLY INORGANIC
GO TO PS 6
EVAPORATES OR NO APPARENT RESPONSE (SUBLIMES)
RESIDUE (DOES NOT BURN)
GO TO PS 6
NO RESIDUE
CONDUCT SPOON FLAME TEST
RESIDUE REMAINS OR NO BURNING
LIQUID H_2O
GO TO PS 6
LIQUID NOT H_2O
GO TO PS 4
SOLID
GO TO PS 6

Figure 4. Procedure set 3 -- flame test.

Results (observations) upon ignition can provide additional insight regarding the composition of waste materials. In general, observations upon ignition can be characterized by one of the following descriptions:

- Burns violently, sparks, sputters, or explodes
- Burns
  - -- Produces very bright (colored) flame
  - -- Burns with a blue flame leaving no smoke or sooty residue
  - -- Burns with yellow flame and produces black smoke and/or sooty residue
  - -- Burns leaving light-colored residue
- Imparts a color to the flame but does not burn
- Melts or fuses, but does not burn
- Evaporates (sublimes) but does not burn

Materials that react violently upon ignition (sputter, spark, explode, or burn with violence) are characterized as explosive (RGN 102).

Waste materials that burn are generally classified as organic except those that give exceptionally bright or colored flames (active free metals -- RGN's 21, 22, 23, and 24). Materials that burn with a yellow flame and produce smoke and/or leave a sooty residue are classifed as organic. This behavior is indicative of organic materials with heteroatoms other than oxygen or aromatic materials. Materials that burn with a nonluminous (blue) flame and produce no smoke or residue are classified as organic. This behavior is characteristic of aliphatic hydrocarbons or materials that contain oxygen as the only heteroatom.

Materials that render a flame colored but do not burn are classified as inorganic. (This behavior is highly characteristic of inorganic salts.) The color of the flame can be indicative of the cations present. In addition, solid materials that fuse but do not burn or evaporate are classified as inorganic.

Solids that produce no apparent response upon ignition are classified as inorganic. Liquids that evaporate but leave no residue are either water solutions of volatile materials (RGN 106) or perhaps chlorinated organic solvents. The results of PS 2 are consulted to distinguish these two possibilities.

Flame tests are conducted primarily to classify materials as organic or inorganic. In addition, more specific indicators are also given regarding the composition of organic waste materials. While the observations to be made in the flame test are somewhat subjective, it is anticipated that most observers can be trained in less than 1 hr to identify explosives and distinguish between organic and inorganic waste materials in a vast majority of real-world situations.

3.3.4 PS 4 -- Sodium Fusion and Ferrox Tests

The classification of material as primarily organic or inorganic is made in PS 2 or 3. PS 4 is conducted if it is determined that the material is organic, and provides elemental analysis information. The flow diagram for PS 4 is shown in Figure 5.

This procedure set consists primarily of two tests: sodium fusion with specific tests for nitrogen (N), sulfur (S), phosphorus (P), and halogen (X); and the ferrox test to identify oxygen (O).

The waste material is first subjected to fusion with sodium metal. The waste material is thus decomposed, and heteroatoms form sodium sulfide (sulfur), sodium cyanide (nitrogen), sodium phosphate (phosphorus) and/or sodium halide (halogen). Separate tests are performed on the fusion extract for sulfide, cyanide, and phosphate. In the presence of sulfur, the thiocyanate test is also performed for nitrogen. In the presence of nitrogen, sulfur, or phosphorus, oxygen is assumed to be present. Organic sulfides, thiazoles, and hydrazides are an exception to this assumption. Appropriate functionality tests are performed if nitrogen (PS 5C) or sulfur (PS 5D) are determined to be present in the sodium fusion procedure. If nitrogen, sulfur, or phosphorus are present, tests for functional groups containing oxygen (PS 5B) are also performed. Organic waste materials containing halogen are classified as halogenated organics (RGN 17).

If nitrogen, sulfur, and/or phosphorus are not detected by the sodium fusion procedure, the ferrox test for organic oxygen compounds is performed. This procedure is applied in the presence or absence of halogen. Appropriate functionality tests for organic oxygen compounds (PS 5B) are performed if the ferrox test is positive. If the ferrox test is negative, the waste material is classified as a hydrocarbon (RGN 16, 28, or 29) or as a halogenated organic (RGN 17). The latter four reactivity groups are mutually exclusive, so RGN's 16, 28, and 29 are not considered in the presence of halogen. In the absence of halogen, optional tests are performed (PS 5A see section 3.3.5.1) to classify the hydrocarbon waste. If the identification of the potential presence of PCB's is desirable then the Friedel-Crafts test should be performed when halogens are present.

Two reactivity groups are determined directly in PS 4, namely organophosphates, phosphothioates, and phosphodithioates (RGN 32) and halogenated organics (RGN 17). Organic waste materials containing phosphorus are classified into RGN 32, since by definition this reactivity group contains most organic waste materials that contain phosphorus. (It is likely that any other organic phosphorus materials would have compatibility characteristics similar to those materials defined by RGN 32.) Halogenated organics are classified into RGN 17 by definition if the fusion procedure indicates the presence of halogen.

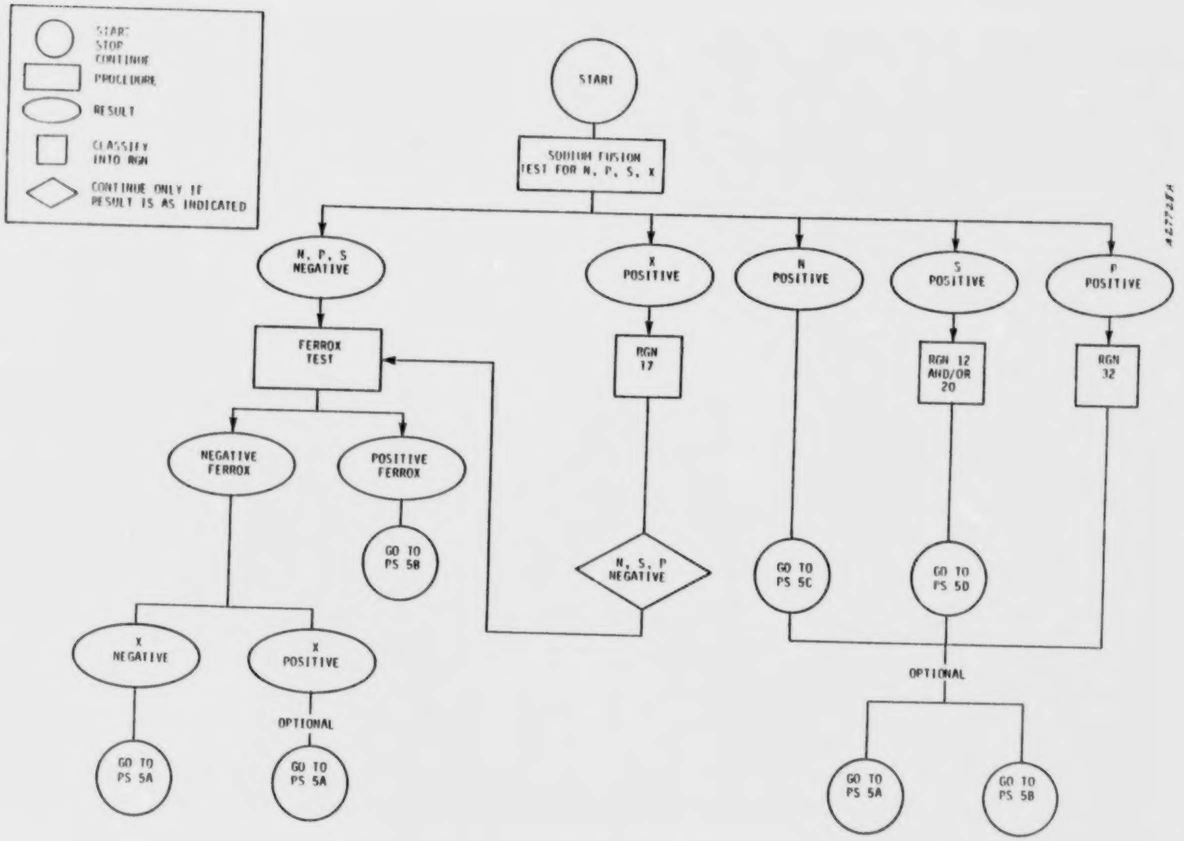

START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY INTO RGN
CONTINUE ONLY IF RESULT IS AS INDICATED
START
SODIUM FUSION TEST FOR N, P, S, X
N, P, S NEGATIVE
X POSITIVE
N POSITIVE
S POSITIVE
P POSITIVE
FERROX TEST
RGN 17
RGN 12 AND/OR 20
RGN 32
NEGATIVE FERROX
POSITIVE FERROX
GO TO PS 5B
N, S, P NEGATIVE
GO TO PS 5C
GO TO PS 5D
X NEGATIVE
X POSITIVE
OPTIONAL
GO TO PS 5A
GO TO PS 5A
OPTIONAL
GO TO PS 5A
GO TO PS 5B

Figure 5. Procedure set 4 -- sodium fusion and ferrox tests.

In summary, PS 4 provides for the elemental analysis of materials determined to be primarily organic in PS 2 or 3. Results of PS 4 are used to determine the sequence of tests to be performed in PS 5.

3.3.5 PS 5 -- Organic Functionality Tests

PS 5 consists of functional group tests for classification of organic waste materials. This procedure set consists of four major parts:

- PS 5A -- Hydrocarbon classification tests (optional)
- PS 5B -- Tests for organic functional groups containing oxygen
- PS 5C -- Tests for organic functional groups containing nitrogen
- PS 5D -- Tests for organic functional groups containing sulfur (optional)

PS 5 includes specific tests for classification of waste materials into 20 RGN's and is outlined in Figures 6a through 6d. The functional groups determined in PS 5 are:

- PS 5A -- Hydrocarbons
    - RGN 16: Hydrocarbons, aromatic
    - RGN 28: Hydrocarbons, aliphatic, unsaturated
    - RGN 29: Hydrocarbons, aliphatic, saturated
- PS 5B -- Oxygen functional groups
    - RGN 3: Organic acids
    - RGN 4: Alcohols and glycols
    - RGN 5: Aldehydes
    - RGN 13: Esters
    - RGN 14: Ethers
    - RGN 19: Ketones
    - RGN 31: Phenols and cresols
    - RGN 34: Epoxides
    - RGN 30: Peroxides
- PS 5C -- Nitrogen functional groups
    - RGN 6: Amides
    - RGN 7: Amines
    - RGN 8: Azo compounds, diazo compounds, and hydrazines
    - RGN 9: Carbamates
    - RGN 18: Isocyanates
    - RGN 26: Nitriles
    - RGN 27: Organic nitro compounds

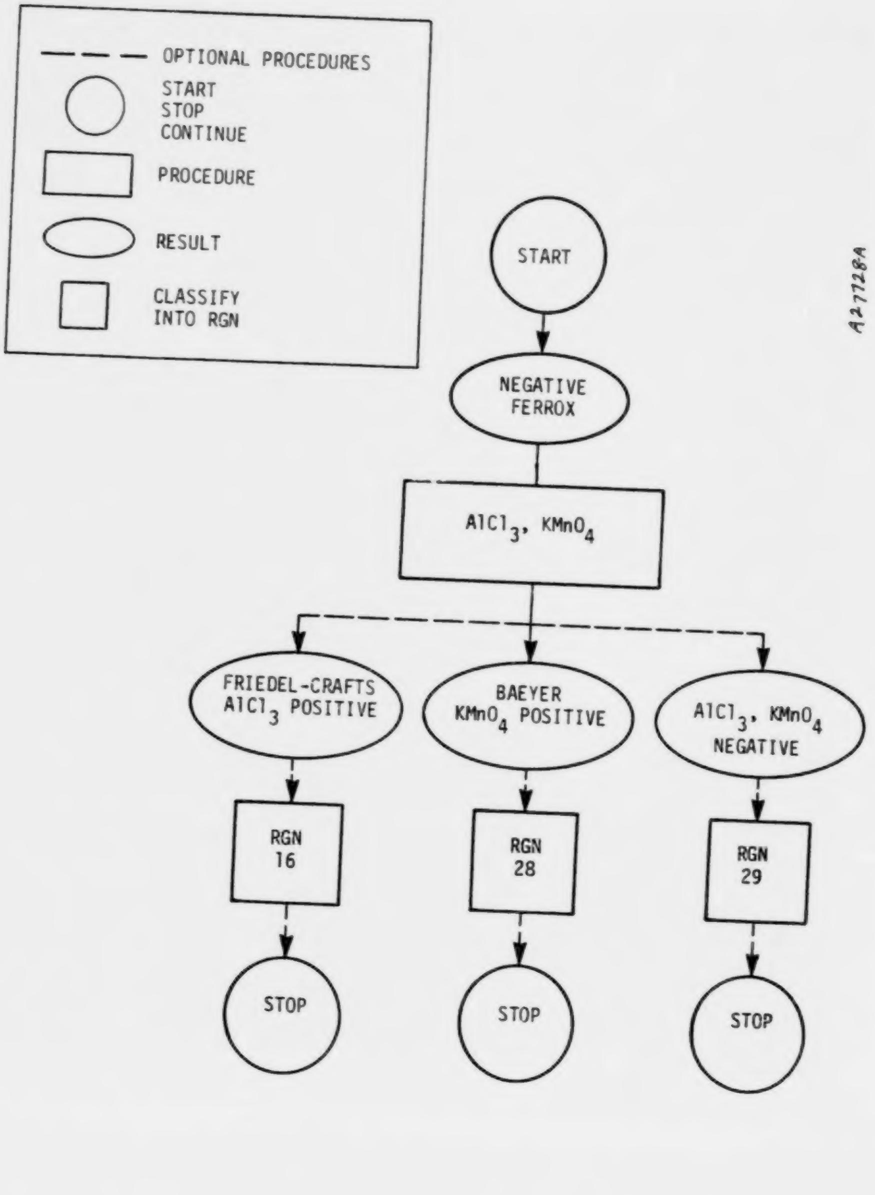

OPTIONAL PROCEDURES
START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY INTO RGN
START
NEGATIVE FERROX
AlCl3, KMnO4
FRIEDEL-CRAFTS AlCl3 POSITIVE
BAEYER KMnO4 POSITIVE
AlCl3, KMnO4 NEGATIVE
RGN 16
RGN 28
RGN 29
STOP
STOP
STOP
A27728A

Figure 6a. Procedure set 5A -- organic functionality tests for hydrocarbon classification.

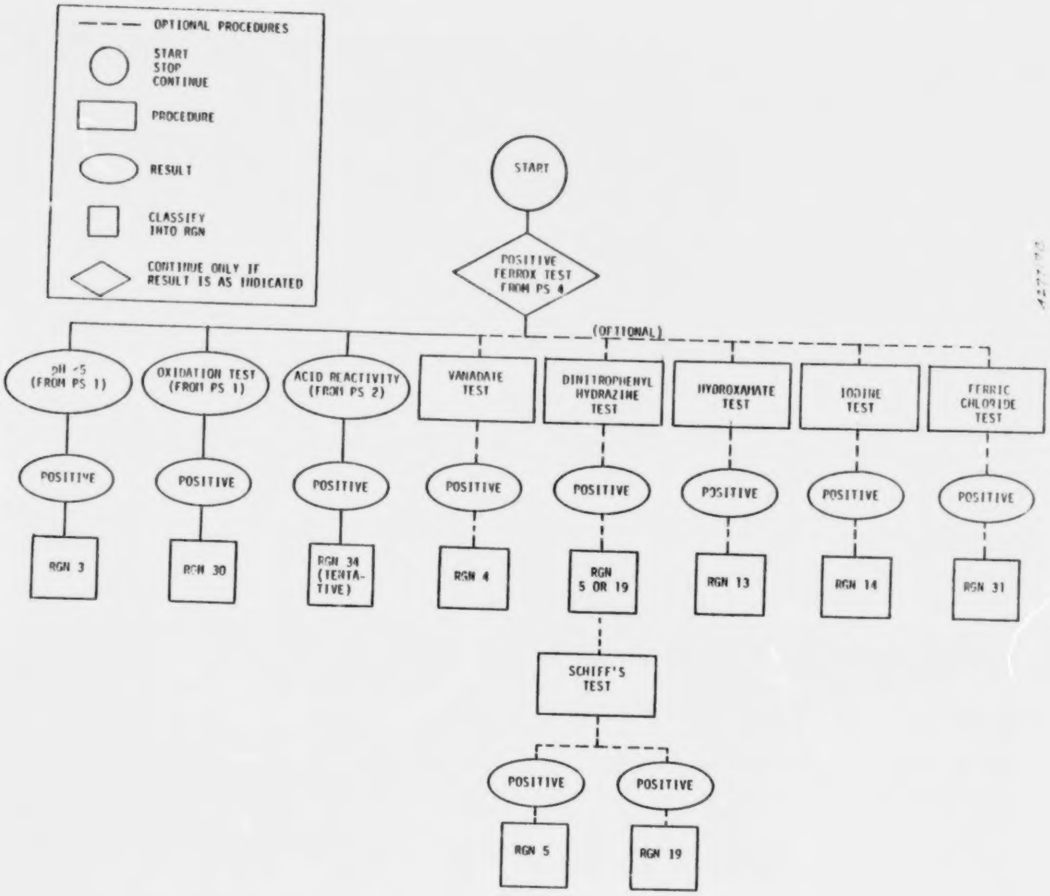

OPTIONAL PROCEDURES
START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY
INTO RGN
CONTINUE ONLY IF
RESULT IS AS INDICATED
START
POSITIVE
FERROX TEST
FROM PS 4
(OPTIONAL)
pH <5
(FROM PS 1)
OXIDATION TEST
(FROM PS 1)
ACID REACTIVITY
(FROM PS 2)
VANADATE
TEST
DINITROPHENYL
HYDRAZINE
TEST
HYDROXAMATE
TEST
IODINE
TEST
FERRIC
CHLORIDE
TEST
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
RGN 3
RGN 30
RGN 34
(TENTA-
TIVE)
RGN 4
RGN
5 OR 19
RGN 13
RGN 14
RGN 31
SCHIFF'S
TEST
POSITIVE
POSITIVE
RGN 5
RGN 19

Figure 6b. Procedure set 5B -- organic functionality tests for functional groups containing oxygen.

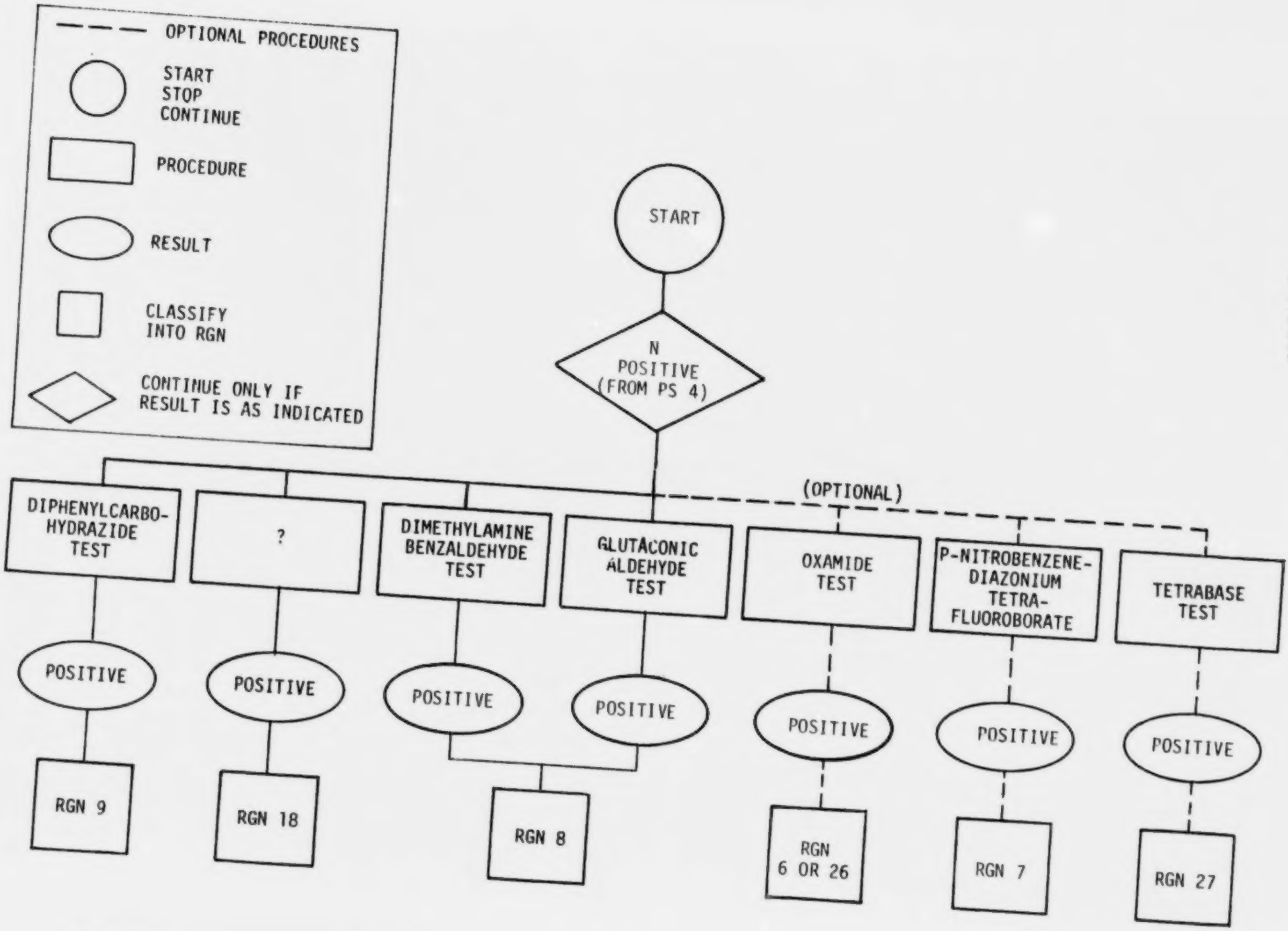
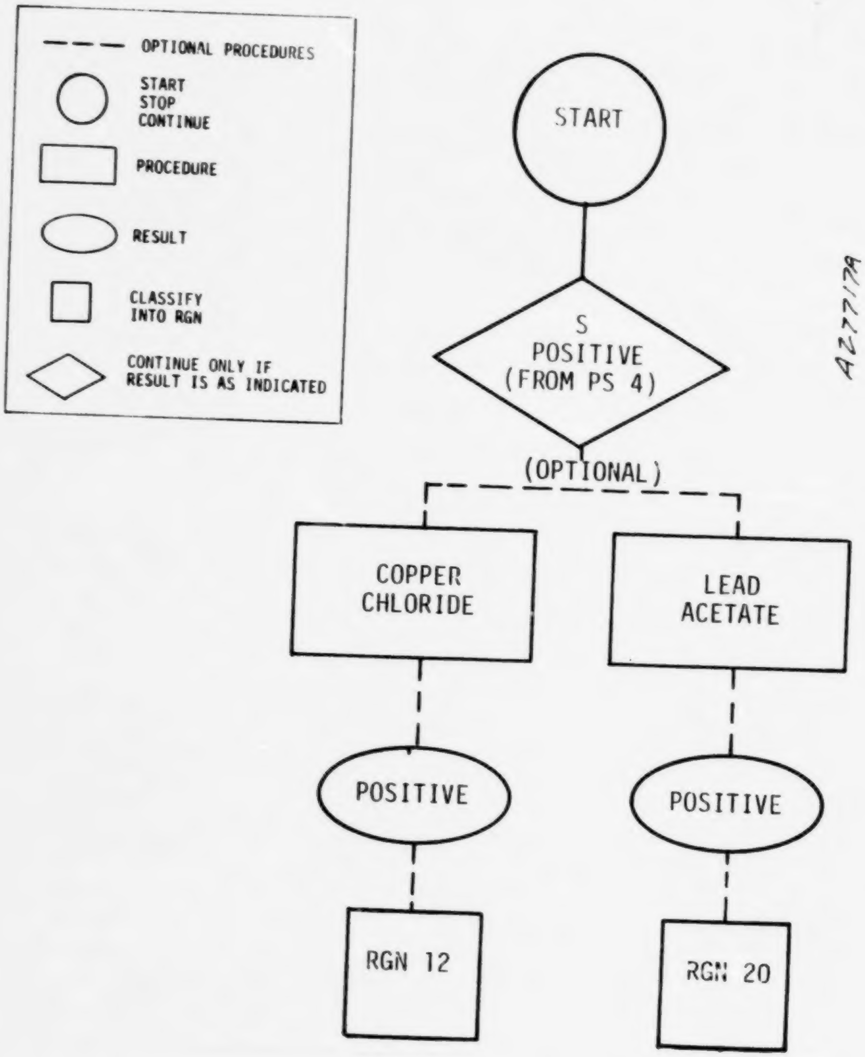

OPTIONAL PROCEDURES
START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY
INTO RGN
CONTINUE ONLY IF
RESULT IS AS INDICATED
START
N
POSITIVE
(FROM PS 4)
(OPTIONAL)
DIPHENYLCARBO-
HYDRAZIDE
TEST
?
DIMETHYLAMINE
BENZALDEHYDE
TEST
GLUTACONIC
ALDEHYDE
TEST
OXAMIDE
TEST
P-NITROBENZENE-
DIAZONIUM
TETRA-
FLUOROBORATE
TETRABASE
TEST
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
RGN 9
RGN 18
RGN 8
RGN
6 OR 26
RGN 7
RGN 27

Figure 6c. Procedure set 5C -- organic functionality tests for functional groups containing nitrogen.

A27717-8


OPTIONAL PROCEDURES
START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY INTO RGN
CONTINUE ONLY IF RESULT IS AS INDICATED
START
S POSITIVE (FROM PS 4)
(OPTIONAL)
COPPER CHLORIDE
LEAD ACETATE
POSITIVE
POSITIVE
RGN 12
RGN 20

A277179

Figure 6d. Procedure set 5D -- organic functionality tests for functional groups containing sulfur.

- PS 5D -- Sulfur functional groups
  - -- RGN 12: Dithiocarbamates
  - -- RGN 20: Mercaptans and other organic sulfides

Each of the procedure subsets 5B to 5D is performed only if the respective element is determined to be present in PS 4. If the element was found to be absent in PS 4, it can be concluded that all reactivity groups determined in that subset are absent. If sulfur, nitrogen, or phosphorus are present, oxygen is also assumed to be present, and PS 5B is conducted in addition to the appropriate procedure subset. If oxygen, nitrogen, halogen, sulfur, and phosphorus are all found to be absent, it is concluded that the material is hydrocarbon and PS 5A is conducted. If any of the elements listed above are present, the three hydrocarbon RGN's are assumed to be absent. The Friedel-Crafts test may be conducted even if heteroatoms are present to identify the aromatic structure. Each of the individual procedure subsets are explained in the following paragraphs.

3.3.5.1 PS 5A -- Tests for Hydrocarbon Classification (Optional)

Three classes of hydrocarbons (RGN's 16, 28, and 29) are distinguished by the Friedel-Crafts test and the Baeyer test. The flow diagram for PS 5A is shown in Figure 6d. These three RGN's are mutually exclusive (by definition); therefore, if one test result is positive, the other test logically need not be run. However, it should be noted that some compounds (e.g., styrene) have both aromatic (RGN 16) and unsaturated aliphatic (RGN 28) functionality. It is suggested that either both tests be performed or the Friedel-Crafts test (RGN 16) be performed first in this sequence. RGN 16 is the more serious category from a toxicity criteria. If both the Friedel-Crafts test and the Baeyer test are negative, the material is classified as RGN 29.

Since the hydrocarbon RGN's are similar in their compatibility characteristics and are all relatively unreactive, this procedure subset is labeled optional.

It should be noted that while it is likely that hydrocarbons may go undetected in mixtures containing other more reactive organics, the compatibility characteristics of any waste material will be dominated by the characteristics of the more reactive material.

3.3.5.2 PS 5B -- Tests for Organic Functional Groups Containing Oxygen

Tests for functional groups containing oxygen involve tests for nine specific reactivity groups. These tests are performed on all organic wastes except those materials shown not to contain sulfur, nitrogen, or phosphorus by the fusion procedure and those that give a negative ferrox test. The flow diagram for procedure subset 5B is shown in Figure 6b. If the ferrox test is negative, all the RGN's in PS 5B are assumed to be absent.

Three of the reactivity groups are identified by means of results obtained in PS 1 and PS 2. Organic acids (RGN 3) are identified by means of their pH (pH 1.5 to 4.5). Organic peroxides (RGN 30) respond as oxidizing agents and should be identified as RGN 104 in PS 1. The result is confirmed as peroxide by the EM Quant® peroxide test strip in this procedure set. Note that this confirmation would take place only if optional testing of materials classified as RGN 104 were conducted. No specific test was found for identification of epoxides (RGN 34), but this reactivity group is tentatively identified by its vigorous, sometimes violent reaction with mineral acids, particularly nitric acid.

The remaining six RGN's identified in PS 5B are less reactive than RGN's 3, 30, or 34. If one of these three reactive RGN's are identified, it is advisable to omit further tests for other RGN's. Furthermore, compatibility characteristics of the less reactive RGN's are similar, so tests for the remaining six RGN's are labeled optional.

Dinitrophenylhydrazine is used to identify carbonyl compounds (aldehydes and ketones, RGN's 5 and 19), and the Schiff's test is used to distinguish between the two. Separate tests are conducted to identify alcohols (RGN 4), esters (RGN 13), ethers (RGN 14), and phenols (RGN 31).

3.3.5.3 PS 5C -- Tests for Organic Functional Groups Containing Nitrogen

Functional group tests for organic compounds containing nitrogen are contained in PS 5C. These tests are performed only if nitrogen is detected by the sodium fusion procedure (PS 4). The flow diagram for classification of the seven reactivity groups containing nitrogen is shown in Figure 6c. If nitrogen is absent, as determined by the sodium fusion procedure (PS 4), all the RGN's in PS 5C are thus determined to be absent.

Three reactivity groups are considerably more reactive than others: carbamates (RGN 9), isocyanates (RGN 18), and azo compounds, diazo compounds, and hydrazines (RGN 8). The diphenylcarbohydrazide test is conducted to identify RGN 9. Azo compounds are identified by the dimethylaminobenzaldehyde test and hydrazines are identified by the glutaconic aldehyde test. If either of these tests are positive, the material is classified as RGN 8. No suitable test procedure has been identified for isocyanates (RGN 18).

If a material is classified as RGN 8, 9, or 18, the need for further testing in PS 5C is eliminated. Tests for RGN's 6, 7, 26, and 27 are included, but are labeled optional, because compatibility characteristics of these materials are similar and the reactivity hazard of these materials is not as great. Amides and nitriles are not distinguished by the test procedure, but these materials are very similar in their reactivity properties.

3.3.5.4 PS 5D -- Tests for Organic Functional Groups Containing Sulfur (Optional)

Tests for two reactivity groups that contain sulfur are included in PS 5D. Since the reactivity characteristics of these RGN's are similar, the procedure subset is labeled optional. If sulfur is shown to be absent in the sodium fusion procedure (PS 4), both RGN's 12 and 20 are therefore determined to be absent. The flow diagram for procedure subset 5D is shown in Figure 6d.

3.3.6 PS 6 -- Inorganic Functionality Tests

If it is determined from PS 2 or 3 that a material is inorganic, three test procedures are conducted to identify inorganic materials not identified in previous procedure sets (PS 1 and 2). Inorganic materials detected in PS 1 and PS 2 are the following:

- RGN 1: Acids, mineral, nonoxidizing
- RGN 2: Acids, mineral, oxidizing
- RGN 10: Caustics
- RGN 11: Cyanides
- RGN 33: Sulfides, inorganic
- RGN 104: Oxidizing agents, strong
- RGN 105: Reducing agents, strong
- RGN 106: Water and mixtures containing water

Tests for remaining inorganic RGN's (15, 21, 22, 23, 24) comprise PS 6, outlined in Figure 7.

Elemental metals or alloys can often be identified visually, but are identified by the phosphomolybdic acid test. Alkali and alkaline earth metals (RGN 21) react with water to give solutions that are basic (PS 2). Visual inspection can be used to identify the form of other elemental metals (RGN's 22 or 23).

It should be noted that all elemental metals except mercury (which is readily identified visually) are solids, so the phosphomolybdic acid test is not conducted if the waste material under investigation is a liquid.

Solid materials are dissolved (using concentrated nitric acid if necessary). This solution or liquid waste material is treated with zinc sulfide to determine the presence of heavy metals, which are assumed to be toxic (RGN 24). Inorganic fluorides (RGN 15) are identified by means of the zirconium alizarinate test.

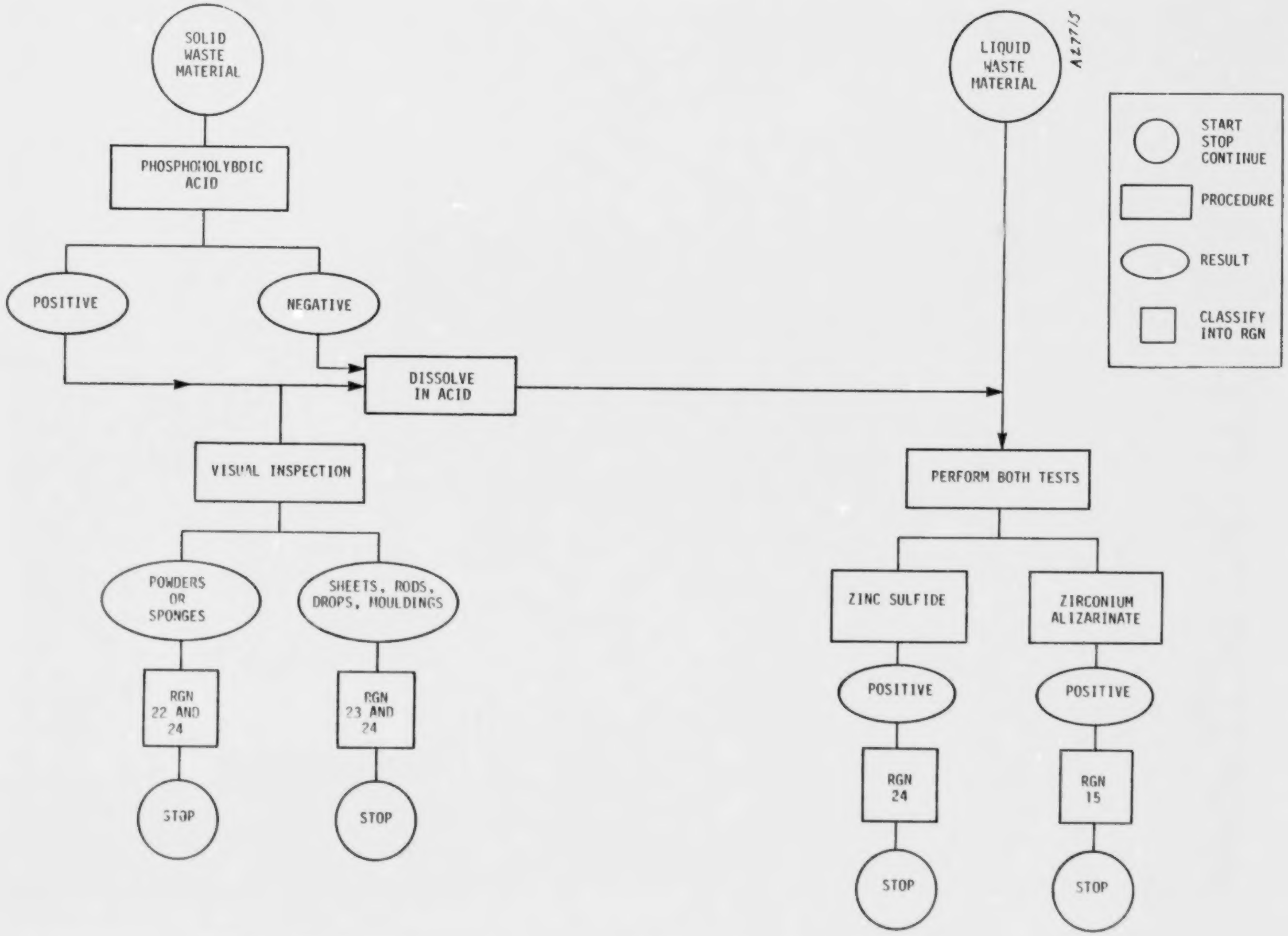

SOLID WASTE MATERIAL
PHOSPHOMOLYBDIC ACID
POSITIVE
NEGATIVE
DISSOLVE IN ACID
VISUAL INSPECTION
POWDERS OR SPONGES
SHEETS, RODS, DROPS, MOULDINGS
RGN 22 AND 24
RGN 23 AND 24
STOP
STOP
LIQUID WASTE MATERIAL
A27715
PERFORM BOTH TESTS
ZINC SULFIDE
ZIRCONIUM ALIZARINATE
POSITIVE
POSITIVE
RGN 24
RGN 15
STOP
STOP
START STOP CONTINUE
PROCEDURE
RESULT
CLASSIFY INTO RGN

Figure 7. Procedure set 6 -- inorganic functionality tests.

It should be noted that a test is not included for nitrides (RGN 25), but these materials are explosive and are thus identified in PS 3. (Nitrides are not available commercially and were therefore not included as part of this study).

3.4 RESULTS OF TESTS USED TO VERIFY PROCEDURE SETS

3.4.1 PS 1 -- pH and Redox Tests

The 58 reference compounds (see Table 2) were tested for oxidation/reduction potential and for pH using test papers. Oxidizing agents were identified by means of starch-iodide test paper (Fisher). Reducing agents were identified in a similar manner with methylene blue or 2,4-dichloroindophenol test paper. The pH of reference compounds was determined using colorpHast® indicator (Merck).

The results of testing the reference compounds using the three test paper procedures above are described in Table 3. No anomalous results were obtained.

Merck pH test strips and Fisher iodide-starch paper were chosen for use in the test kit because they are commercially available, reliable, and easy to use. Methylene blue test strips were easy to use, reliable, and easy to prepare.

3.4.2 PS 2 -- Solution Reactivity and Special Functionality Tests

3.4.2.1 Solution Reactivity Tests

The reference compounds were treated with water, a series of mineral acids and bases, and four organic solvents to assess solubility and/or reactivity characteristics. These characteristics can give clues regarding the general chemical characteristics of the waste material. In addition, these tests quickly predict the compatibility of the water material with other waste materials likely to be found at a hazardous waste disposal site.

While the materials can be categorized according to solubility and reactivity by this set of spot tests, the utility of the information derived is limited. The information is of general utility in handling the waste materials. Those materials found to be soluble in hexane and/or toluene can be classified as organic. Likewise, materials found to be soluble in acetone and/or methanol but not in water can be considered organic. Most water-reactive materials can be identified by means of this drop test.

The results of solution-reactivity tests conducted on the reference compounds are presented in Table 4. None of the observations represent a deviation from those expected. The results of these tests are correlated by grouping the test materials according to general chemical class (i.e., elemental metal, hydrocarbon, organic oxygen compound) and noting the response of all the reference compounds of that particular general class. A

TABLE 3. RESPONSE OF REFERENCE COMPOUNDS TO pH AND REDOX TESTS

Compound	RGN(s)	Response[a] pH	Oxidizing	Reducing
Chromic oxide	2,24,104	0	+	-[b]
Cadmium	23,24	NR	NR	NR
Sodium	25,105,107	14		
Arsenic pentasulfide	24,33	6	NR	NR
Chromium	23,24	NR	NR	NR
Copper	23,24	NR	NR	NR
Lead	23,24	NR	NR	NR
Nickel	22,24	NR	NR	NR
Barium	21,24,107	14		
Barium iodide	24	5	+	NR
Barium oxide	10,24,107	14	NR	NR
Calcium hypochlorite	10,104	9	+	NR
Selenium diethyldithiocarbamate	12,24	7	NR	NR
Hydrofluorosilicic acid	1,15	0	+	NR
Peracetic acid	3,30,104	2	+	NR

(continued)

[a] NR = no response
+ = positive test result
- = negative test result

[b] The test paper was decolorized (bleached) by the strong oxidizing agent. Results are clear when test material is diluted with water.

TABLE 3. (continued)

Compound	RGN(s)	Response[a]		
		pH	Oxidizing	Reducing
Fluoboric acid	1,15	0	+	NR
Hexafluorophosphoric acid	1,15	0	+	NR
Potassium cyanide	11	12	NR	NR
Hydrofluoric acid	1,15	0	NR	NR
Trinitrobenzoic acid	3,27,102	0	NR	NR
Picric acid	27,31,102	2	NR	NR
Dipicrylamine	27,31,102	6	NR	NR
Dimethylaminoazobenzene	7,8	NR	NR	NR
Aminopropionitrile	7,26	11	NR	NR
Aminothiazole	7	7	NR	NR
Benzoyl peroxide	30,102	NR	+	NR
Malonic nitrile (cyanoacetic acid)	3,26	2	NR	NR
Toluene diisocyanate	18,107	NR	NR	NR
Hydroxylamine hydrochloride	105	NR	NR	+

(continued)

[a] NR = no response
+ = positive test response
- = negative test response

[b] The test paper was decolorized (bleached) by the strong oxidizing agent. Results are clear when test material is diluted with water.

TABLE 3. (continued)

Compound	RGN(s)	Response[a] pH	Oxidizing	Reducing
Malathion	13,32	NR	NR	NR
Parathion	27,32	NR	NR	NR
Ethyl zimate	12,24	NR	NR	NR
Vinyl acetate	13,103	NR	NR	NR
Bromoxynil	13,17,26	NR	NR	NR
Chlorophenyl isocyanate	17,18,107	NR	NR	NR
Styrene	16,28,103	NR	NR	NR
Ethylene glycol monomethyl ether	4,14	NR	NR	NR
Polypropylene	29,101	NR	NR	NR
Propylene oxide	34,103	NR	NR	NR
Methyl methacrylate	13,103	NR	NR	NR
Ethyl acrylate	13,103	NR	NR	NR
Mercaptobenzothiazole	20	NR	NR	NR
Lannate	9	NR	NR	NR
Hydroxyacetophenone	19,31	NR	NR	NR

[a] NR = no response
+ = positive test response
- = negative test response

(continued)

[b] The test paper was decolorized (bleached) by the strong oxidizing agent. Results are clear when test material is diluted with water.

TABLE 3. (concluded)

Compound	RGN(s)	Response[a]		
		pH	Oxidizing	Reducing
Mercaptoethanol	4,20	NR	NR	+
n-Butyl acrylate	13,103	NR	NR	NR
Acrolein	5,103	NR	NR	NR
Diacetone alcohol	4,19	NR	NR	NR
Aldicarb	9	NR	NR	NR
Zineb	12,24	NR	NR	NR
Diphenamide	6	3	NR	NR
Hexene	28	NR	NR	NR
Decane	28	NR	NR	NR
TEPA	7,32	7	NR	NR
Acetamide	6	5	NR	NR
Naphthalene	16	NR	NR	NR
Polysulfide polymer	20,14,105	NR	NR	+

[a] NR = no response
+ = positive test response
- = negative test response

[b] The test paper was decolorized (bleached) by the strong oxidizing agent. Results are clear when test material is diluted with water.

TABLE 4. RESPONSE OF REFERENCE COMPOUNDS TO SOLUTION-REACTIVITY TESTS

Compound	RGN(s)	Response								
		Water	Hydrochloric acid	Nitric acid	Sulfuric acid	Sodium hydroxide	Methanol	Acetone	Hexane	Toluene
Chromic oxide	2,24,104	Dissolves readily, turns orange	Dissolves, turns brown	Dissolves slightly	Dissolves slightly	Dissolves readily	Violent reaction, turns black	Violent reaction, turns black	NR[a]	NR
Cadmium	23,24	NR	Dissolves with gas evolution	Violent reaction, brown fumes	Slow reaction (gas evolution)	NR	NR	NR	NR	NR
Sodium/oil dispersion	21,105,107	Slow gas evolution	Slow gas evolution	Slow gas evolution	Slow gas evolution	Gas evolution	Gas evolution	Gas evolution	NR	NR
Arsenic pentasulfide	24,33	Dissolves slowly	Dissolves slowly	Dissolves slowly	Dissolves slowly	Dissolves slowly	NR	NR	NR	NR
Chromium	23,24	NR	Gas evolution	NR	Slow gas evolution	NR	NR	NR	NR	NR
Copper	23,24	NR	Slow gas evolution	Fast gas evolution, brown fumes	NR	NR	NR	NR	NR	NR
Lead	23,24	NR	Slow gas evolution	Slow gas evolution	NR	NR	NR	NR	NR	NR
Nickel	22,24	NR	Slow gas evolution	Gas evolution, brown fumes	Very slow gas evolution	Gas evolution	Gas evolution	Gas evolution	NR	NR
Barium	21,24,107	Fast gas evolution	Fast gas evolution	Fast gas evolution, brown fumes	Fumes, black residue	Dissolves	Dissolves	Dissolves	NR	NR
Barium iodide	24	Dissolves	Dissolves	Fumes, black residue	Dissolves	Dissolves very slowly	NR	NR	NR	NR
Barium oxide	10,24,107	Dissolves very slowly	Gas evolution	Gas evolution	Fast gas evolution	NR	NR	NR	NR	NR
Calcium hypochlorite	10,104	Dissolves, fumes	Fast gas evolution	Fast gas evolution	Turns brown	NR	Dissolves	Dissolves	NR	NR

[a]NR = no observable response

(continued)

TABLE 4. (continued)

Compound	RGN(s)	Water	Hydrochloric acid	Nitric acid	Sulfuric acid	Sodium hydroxide	Methanol	Acetone	Hexane	Toluene
		Response								
Selenium diethyldithiocarbamate	12,24	NR[a]	NR	Brown fumes	Gas evolution	Dissolves	Dissolves	NR	HR	Dissolves
Hydrofluorosilicic acid	1,15	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves	NR	NR
Peracetic acid	3,30,104	Dissolves	Dissolves	Dissolves	Fumes	Dissolves	Dissolves	NR	NR	NR
Fluoboric acid	1,15	Dissolves	Dissolves	Dissolves	Fumes	Dissolves	Dissolves	Dissolves	NR	NR
Hexafluorophosphoric acid	1,15	Dissolves	Dissolves	Dissolves	Fumes	Dissolves	Dissolves	Dissolves	NR	NR
Potassium cyanide	11	Dissolves	Fumes	Fumes	Fumes	Dissolves	NR	NR	NR	NR
Hydrofluoric acid	1,15	Dissolves	Dissolves	Dissolves	Fumes	Dissolves	Dissolves	Dissolves	NR	NR
Trinitrobenzoic acid	3,37,102	Dissolves	Dissolves slowly	Dissolves	Dissolves	Dissolves, turns red	Dissolves	Dissolves	NR	Dissolves very slowly
Picric acid	27,31,102	Dissolves	Dissolves	Dissolves	Dissolves	Turns orange	Dissolves	Dissolves	Dissolves	Dissolves
Dipicrylamine	27,31,102	Dissolves slowly	Dissolves slowly	Dissolves slowly	Dissolves slowly	Turns dark red	Dissolves	Dissolves	NR	NR
Dimethylaminoazobenzene	7,8	NR	Dissolves, turns red	Dissolves, turns red then black	NR	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves
Aminopropionitrile	7,26	Dissolves	White fumes	White fumes	White fumes, viscous residue	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves
Aminothiazole	7	NR	Dissolves, turns brown	Dissolves, turns brown	Dissolves. turns brown	NR	Dissolves	Dissolves	NR	NR
Benzoyl peroxide	30,102	NR	NR	NR	Fumes, turns black	NR	NR	Dissolves slowly	NR	Dissolves slowly

[a] NR = no observable response

(continued)

TABLE 4. (continued)

Compound	RGN(s)	Response								
		Water	Hydrochloric acid	Nitric acid	Sulfuric acid	Sodium hydroxide	Methanol	Acetone	Hexane	Toluene
Malonic nitrile (cyanoacetic acid)	3,26	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves	NR	NR
Toluene diisocyanate	18,107	NR	NR	Gas evolution, turns brown	Gas evolution, white residue	NR	NR	Dissolves	Dissolves	Dissolves
Hydroxylamine hydrochloride	105	Dissolves slowly	Dissolves slowly	Dissolves	Foaming	Dissolves	Dissolves	Dissolves slowly	NR	NR
Malathion	13,32	NR	Dissolves	Gas evolution, turns brown	Gas evolution, turns yellow	NR	Dissolves	Dissolves	Dissolves	Dissolves
Parathion	27,32	NR	Dissolves, turns gray	Turns brown	Dissolves, turns yellow	Turns yellow	Dissolves	Dissolves	Dissolves	Dissolves
Ethyl zimate	12,24	NR	Dissolves turns gray	Gas evolution, brown fumes	Gas evolution	NR	Dissolves	Dissolves	NR	Dissolves
Vinyl acetate	13,103	Dissolves	Dissolves	Dissolves	Turns brown	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves
Bromoxynil	13,17,26	White emulsion	Turns brown	Turns brown	Turns brown	Turns brown	NR	Dissolves	Dissolves	Dissolves
Chlorophenyl isocyanate	17,18,107	NR	NR	Turns brown	Turns brown	NR	Dissolves	Dissolves	Dissolves slowly	Dissolves
Styrene	16,28,103	NR	Dissolves	Dissolves	Turns brown	NR	Dissolves	Dissolves	Dissolves	Dissolves
Ethylene glycol monomethyl ether	4,14	Dissolves	Dissolves	Dissolves	Dissolves turns yellow	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves
Propypropylene	29,101	NR[a]	NR	NR	NR	NR	NR	NR	NR	NR
Propylene oxide	34,103	Gas evolution	Dissolves, gas evolution	Violent spattering	Violent spattering, turns black	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves

[a] NR = no observable response

(continued)

TABLE 4. (concluded)

Compound	RGN(s)	Response								
		Water	Hydrochloric acid	Nitric acid	Sulfuric acid	Sodium hydroxide	Methanol	Acetone	Hexane	Toluene
Methyl methacrylate	13,103	NR	Dissolves	Dissolves	Dissolves	NR	Dissolves	Dissolves	Dissolves	Dissolves
Ethyl acrylate	13,103	NR	Dissolves	Dissolves	Dissolves	NR	Dissolves	Dissolves	Dissolves	Dissolves
Mercaptobenzothiazole	20	NR	Dissolves	Gas evolution, brown fumes	Dissolves	Dissolves	NR	Dissolves	NR	Dissolves
Lannate	9	NR	Dissolves	Dissolves	Dissolves	Dissolves slowly	Dissolves	Dissolves	Dissolves	Dissolves
Hydroxyacetophenone	19,31	NR	NR	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves	NR	Dissolves
Mercaptoethanol	4,20	Dissolves	Dissolves	Dissolves, turns pink	Foams	NR	Dissolves	Dissolves	Dissolves	Dissolves
n-Butyl acrylate	13,103	NR	NR	NR	Dissolves	NR	Dissolves	Dissolves	Dissolves	Dissolves
Acrolein	5,103	NR	Dissolves	Dissolves	Turns black	Fumes, white residue	Dissolves	Dissolves	Dissolves	Dissolves
Diacetone alcohol	4,19	Dissolves	Turns orange	Turns orange	Turns brown	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves
Aldicarb	9	NR	Dissolves	Dissolves	Dissolves	NR	Dissolves	Dissolves	Dissolves	Dissolves
Zineb	12,24	NR	Dissolves	Dissolves	Dissolves	NR	Dissolves	Dissolves	Dissolves	Dissolves
Diphenamide	6	NR	Dissolves	Dissolves	Dissolves	NR	Dissolves	Dissolves	Dissolves	Dissolves
Hexene	28	NR	Dissolves	Dissolves	Dissolves	NR	Dissolves	Dissolves	Dissolves	Dissolves
Decene	28	NR	NR	NR	Dissolves	NR	Dissolves	Dissolves	Dissolves	Dissolves
TEPA	7,32	Dissolves	Dissolves	Dissolves slowly	Gas evolution, turns black	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves
Acetamide	6	Dissolves	NR	NR	Dissolves	Dissolves	Dissolves	Dissolves	NR	NR
Naphthalene	16	NR	NR	NR	NR	NR	NR	Dissolves	NR	Dissolves
Polysulfide polymer	14,20,105	NR	NR	Brown fumes	Dissolves	NR	NR	Dissolves	NR	Dissolves

[a] NR = no observable response

generalized summary of the solution reactivity tests is presented in Table 5.

3.4.2.2 Special Functionality Tests

Concurrent with the solution reactivity tests, the reference compounds were tested for the presence of sulfide and cyanide, and liquids were tested for the presence of water. Sulfide is detected using lead acetate test paper, cyanide is determined by a spot test (Table 1), and the presence of water is determined in liquids by anhydrous copper sulfate. These tests are conducted early in the testing sequence to identify these hazards before extensive testing is initiated. Since they involve spot tests, they are conveniently performed concurrently with the solution reactivity tests.

The results of the special functionality tests are presented in Table 6. Sulfide-containing test materials render anhydrous copper sulfate black (by formation of copper sulfide). Aminoproprionitrile produces a violet color, the origin of which is unknown. These were the only anomalous results oberserved in the special functionality tests.

3.4.3 PS 3 -- Flame Tests

The behavior of each of the reference compounds upon ignition was observed by performing the flame test. In most cases, an adequate observation was made using a wire loop. In those cases where observation was not possible using a wire loop, a porcelain spatula was used. The responses of each of the test materials to the flame test are presented in Table 7. Five categories of behavior are presented in Table 7: fusion (melting) of the test material, burning, color of the flame produced, smoke produced during ignition, and any residue remaining after ignition.

A generalized summary of the behavior of the reference compounds upon ignition is presented in Table 8. Most of the test materials respond as predicted, that is, organic materials burn and inorganic materials do not burn. Notable exceptions are elemental metals which burn with characteristically colored flames and oxidizing and reducing agents which generally exhibit vigorous to violent behavior upon ignition. Therefore care must be taken when classifying combustible materials as organic. In particular, one must be sure to consult the results of PS 1 and PS 2, because the behavior of oxidizing and reducing agents in the flame test can be misleading. Solubility information from PS 2 is used in conjunction with the flame test results to classify these materials as organic or inorganic. Three of the four compounds falling into the explosive category (RGN 102) showed distinctive behavior in the flame test. However, trinitrobenzoic acid did not show an exceptional response.

3.4.4 PS 4 -- Sodium Fusion and Ferrox Test

A determination of the presence of heteroatoms in the reference compounds was made using tests in PS 4. Nitrogen, sulfur, chloride, and phosphorus were detected by fusion of the material with metallic sodium and

TABLE 5. GENERAL SUMMARY OF SOLUTION-REACTIVITY TEST RESULTS

	Response						
Compound	Water	Hydrochloric acid	Nitric acid	Sulfuric acid	Sodium hydroxide	Polar solvents methanol, acetone	Nonpolar solvents hexane, toluene
Hydrocarbons	NR[a]	NR	NR	NR	NR	Dissolves, acetone NR, methanol	Dissolves (except polypropylene)
Esters, ethers, acids, aldehydes, and ketones	NR	Dissolves	Dissolves (color change)	Dissolves (turns brown)	Dissolves or NR	Dissolves or NR	Dissolves or NR
Nitro compounds and picrates	Dissolves	Dissolves	Dissolves	Dissolves	Dissolves, red color	Dissolves or NR	Dissolves or NR
Amines	NR or Dissolves	Dissolves	Dissolves (discolored)	NR or darkens	NR	Dissolves or NR	Dissolves or NR
Isocyanates, nitriles	NR	NR	Vigorous reaction, brown fumes	NR or darkens	NR	Dissolves or NR	Dissolves or NR
Mercaptans	NR	NR or dissolves	Brown fumes	NR or darkens	Dissolves or NR	Dissolves or NR	Dissolves or NR
Phosphorus compounds	NR	NR or dissolves	Brown fumes	Darkens	Dissolves or NR	Dissolves or NR	Dissolves or NR
Epoxides	Dissolves, gas	Dissolves, gas	Violent spattering	Viorous reaction	Dissolves or NR	Dissolves or NR	Dissolves or NR
Reactive olefins (polymerizable)	NR	Dissolves	Dissolves or NR	Dissolves or darkens	NR	Dissolves or NR	Dissolves or NR
Inorganic acids	Dissolves	Dissolves	Dissolves	Dissolves, fumes	Dissolves	Dissolves or NR	NR
Metallic elements	NR except Na and Ba (gas evolution)	Slow gas evolution	Gas, brown fumes (some metals violent reaction)	Slow reaction (gas evolution)	NR except Na and Ba (gas evolution)	NR except Na and Ba (gas evolution)	NR
Oxidizing agents	Dissolves	Dissolves, $Ca(OCl)_2$, fumes	Dissolves, $Ca(OCl)_2$, fumes	Dissolves, $Ca(OCl)_2$, fumes	Dissolves	CrO_3, violent $Ca(OCl)_2$, NR	NR
Salts and oxides	Dissolves or NR	Dissolves, fumes, or NR	Dissolves, fumes, or NR	Dissolves, fumes, or NR	Dissolves or NR	NR	NR

[a] NR = No observable response

TABLE 6. RESPONSE OF REFERENCE COMPOUNDS TO THE SPECIAL FUNCTIONALITY TESTS

Compound	RGN(s)	Response[a]		
		CN^-	$S^=$	H_2O[b]
Chromic oxide	2,24,104	-	-	NA
Cadmium	23,24	-	-	NA
Sodium	25,105,107	-	-	NA
Arsenic pentasulfide	24,33	-	+	NA
Chromium	23,24	-	-	NA
Copper	23,24	-	-	NA
Lead	23,24	-	-	NA
Nickel	23,24	-	-	NA
Barium	21,24,107	-	-	NA
Barium iodide	24	-	-	NA
Barium oxide	10,24,107	-	-	NA
Calcium hypochlorite	10,104	-	-	NA
Selenium diethyldithiocarbamate	12,24	-	-	NA
Hydrofluorosilicic acid	1,15	-	-	+
Peracetic acid	3,30,104	-	-	+

(continued)

[a] + = positive test response
- = negative test response

[b] Test only conducted with liquid test materials. Solids are listed as NA, not applicable.

[c] Black copper sulfide is produced with waste materials containing sulfides

TABLE 6. (continued)

Compound	RGN(s)	Response[a]		
		CN^-	$S^=$	H_2O[b]
Fluoboric acid	1,15	-	-	+
Hexafluorophosphoric acid	1,15	-	-	+
Potassium cyanide	11	+	-	NA
Hydrofluoric acid	1,15	-	-	+
Trinitrobenzoic acid	3,27,102	-	-	NA
Picric acid	27,31,102	-	-	NA
Dipicrylamine	27,31,102	-	-	NA
Dimethylaminozaobenzene	7,8	-	-	NA
Aminopropionitrile	7,26	-	-	Turns violet
Aminothiazole	7	-	-	NA
Benzoyl peroxide	30,102	-	-	NA
Malonic nitrile (cyanoacetic acid)	3,26	-	-	NA
Toluene diisocyanate	18,107	-	-	-
Hydroxylamine hydrochloride	105	-	-	NA

[a] + = positive test response
- = negative test response

(continued)

[b] Test only conducted with liquid test materials. Solids are listed as NA, not applicable.

[c] Black copper sulfide is produced with waste materials containing sulfides

TABLE 6. (continued)

Compound	RGN(s)	Response[a]		
		CN^-	$S^=$	H_2O[b]
Malathion	13,32	-	-	-
Parathion	27,32	-	-	-
Ethyl zimate	12,24	-	-	NA
Vinyl acetate	13,103	-	-	-
Bromoxynil	13,17,26	-	-	-
Chlorophenyl isocyanate	17,18,107	-	-	NA
Styrene	16,28,103	-	-	-
Ethylene glycol monomethyl ether	4,14			
Polypropylene	29,101	-	-	NA
Propylene oxide	34,103	-	-	-
Methyl methacrylate	13,103	-	-	-
Ethyl acrylate	13,103	-	-	-
Mercaptobenzothiazole	20	-	-	NA
Lannate	9	-	-	NA
Hydroxyacetophenone	19,31	-	-	NA

[a] + = positive test response
 - = negative test response

(continued)

[b] Test only conducted with liquid test materials. Solids are listed as NA, not applicable.

[c] Black copper sulfide is produced with waste materials containing sulfides

TABLE 6. (concluded)

Compound	RGN(s)	Response[a]		
		CN^-	$S^=$	H_2O[b]
Mercaptoethanol	4,20	-	+	Turns black[b]
n-Butyl acrylate	13,103	-	-	-
Acrolein	5,103	-	-	-
Diacetone alcohol	4,19			
Aldicarb	9	-	-	NA
Zineb	12,24	-	-	NA
Diphenamide	6	-	-	NA
Hexene	28	-	-	-
Decene	28	-	-	-
TEPA	7,32	-	-	NA
Acetamide	6	-	-	NA
Naphthalene	16	-	-	NA
Polysulfide polymer	14,20,105	-	+	Turns black[c]

[a] + = positive test response
- = negative test response

[b] Test only conducted with liquid test materials. Solids are listed as NA, not applicable.

[c] Black copper sulfide is produced with waste materials containing sulfides

TABLE 7. RESPONSE OF REFERENCE COMPOUNDS TO THE FLAME TESTS

Compound	RGN(s)	Response				
		Melt	Burn	Flame	Smoke	Residue
Chromic oxide	2,24,104	No	Yes, sparkles	Bright (white)	--	Green
Cadmium	23,24	No	Yes	Yellow-red	Yellow	Gray
Sodium	24,105,107	No	Yes	Yellow	NC	Gray
Arsenic pentasulfide	24,33	No	Yes	Blue	Gray	Gray
Chromium	23,24	No	Bright sparkles	White	No	Gray
Copper	23,24	No	No	Green	No	Black
Lead	23,24	No	Yes	Light blue	No	Gray
Nickel	22,24	No	No	None observed	No	Gray
Barium	21,24,107	No	Yes	Orange	No	Gray
Barium iodide	24	Yes	No	Orange	No	No
Barium oxide	10,24,107	Yes	No	Orange	No	Colorless
Calcium hypochlorite	10,104	Yes	No	Orange	No	Colorless
Selenium diethyl dithiocarbamate	12,24	No	Yes	Yellow	White	No
Hydrofluoroxilic acid	1,15	(Liquid)	No	Yellow (Na)	No	No
Peracetic acid	3,30	No	Yes	None	White	No
Fluoboric acid	1,15	(Liquid)	No	Bright green	No	No
Hexafluorophosphoric acid	1,15	(Liquid)	Yes	Yellow	No	No
Potassium cyanide	11	Yes	No	Pink	No	No
Hydrofluoric acid	1,15	(Liquid)	No	Yellow (Na)	No	No
Trinitrobenzoic acid	3,27,102	Yes	Yes	Yellow	Black	Black, sooty
Picric acid	27,31,102	Yes	Sparkles	Yellow	Black	Black, sooty
Dipicrylamine	7,27,102	Yes	Yes, sparkles	Yellow	Black	Black, sooty
Aninopropionitrile	7,26	Yes	Yes	Blue-red	White	No
Aminothiazole	7	Yes	Yes	Blue	No	No
Benzoyl peroxide	30,102	No	Violent	Yellow	No	No
Malonic nitrile (cyanoacetic acid)	3,26	Yes	Yes	Yellow	White	No

(continued)

TABLE 7. (concluded)

		Response				
Compound	RGN(s)	Melt	Burn	Flame	Smoke	Residue
Toluene diisocyanate	18,107	(Liquid)	Yes, foams	Yellow	White	Sooty
Hydroxylamine hydrochloride	105	Yes	No	--	--	White
Malathion	13,32	(Liquid)	Yes	Yellow	Black	Black
Parathion	27,32	(Liquid)	Yes	Yellow	Black	Black
Ethyl zimate	12,24	Yes	Yes	Yellow	White	Gray
Vinyl acetate	13,103	(Liquid)	Yes	Blue	No	No
Bromoxynil	17,26,31	(Liquid)	Yes	Yellow	Black	Black
Chlorophenyl isocyanate	17,18,107	(Liquid)	Yes	Yellow	Black	Black
Stryene	16,28,103	(Liquid)	Yes	Yellow	Black	Black
Ethylene glycol monomethyl ether	4,14	(Liquid)	Yes	Blue	No	No
Polypropylene	29,101	Yes	Yes	Yellow	No	Black
Propylene oxide	34,104	(Liquid)	Yes	Yellow	No	No
Methyl methacrylate	13,103	(Liquid)	Yes	Yellow	No	No
Ethyl acrylate	13,103	(Liquid)	Yes	Yellow	No	No
Mercaptobenzothiazole	20	Yes	Yes	Yellow	White	Gray
Lannate	9	Yes	Yes	Yellow	White	Gray
Hydroxyacetaphenone	19,31	Yes	Yes	Yellow	Black	Black, sooty
Mercapthoethanol	4,20	(Liquid)	Yes	Blue	White	Gray
n-Butyl acrylate	13,103	(Liquid)	Yes	Blue	No	No
Acrolein	5,103	(Liquid)	Yes	White, yellow	No	No
Diacetone alcohol	4,19	(Liquid)	Yes	Blue	No	No
Aldicarb	9	Yes	Yes	Bright yellow	No	No
Zineb	12,24	No	No	None observed	White	White
Diphenamide	6	Yes	Yes	Yellow	White	Gray
Hexene	28	(Liquid)	Yes	Yellow	No	No
Decene	28	(Liquid)	Yes	Yellow	No	No
TEPA	7,32	Yes	Yes	Yellow	Black	Sooty
Acetamide	6	Yes	No	--	White	Gray
Naphthalene	16	Yes	Yes	Yellow	Black	Black, sooty

TABLE 8. GENERAL SUMMARY OF FLAME TEST RESULTS

General compound class	Flame test
Metals and metal salts	Colored flames; cadmium, chromium, lead, barium, and sodium burn brightly, others do not burn; CrO_3 burns very brightly
Acids -- inorganic	Generally do not burn, fluoboric acid bright green; most give yellow (sodium) flame
Aliphatic hydrocarbons and oxygen compounds	Benzoyl peroxide burns violently, others have blue flames -- no smoke -- no residue; alkenes give yellow flames
Aromatic hydrocarbons	Yellow flames, black smoke
Organosulfur, phosphorus, and nitrogen compounds	Burns with white-yellow smoke
Explosives (picrates)	Burns violently (sparkles)

analysis of the resulting extract for cyanide (nitrogen), sulfide (sulfur), halide (halogen), and phosphate (phosphorus). If any of these heteroatoms were detected by the fusion procedure, oxygen was also assumed to be present. If nitrogen, sulfur, and phosphorus were determined to be absent by the fusion procedure, oxygen was detected in the waste material by means of the ferrox test. All of the organic test materials were tested for the presence of nitrogen, sulfur, halogen, and phosphorus with the sodium fusion procedure and for the presence of oxygen with the ferrox test. The results of these tests are presented in Table 9.

Two of the test materials, aminothiazole and mercaptobenzathiazole, gave negative cyanide tests. The fusion extracts from these reference compounds were subsequently tested for thiocyanate, which gave positive results for both extracts. Therefore, in the test scheme for materials containing sulfur, nitrogen is detected by both the cyanide and the thiocyanate tests (i.e., in those cases when sulfur is present and the cyanide test is negative, a test for thiocyanate is also performed). No other anomalous results were obtained for the sodium fusion tests.

All of the organic reference compounds were subjected to the ferrox test. Variable results were obtained for compounds containing nitrogen and sulfur, so this test cannot be used alone to detect oxygen. For example, organic nitro compounds give a negative response and aminothiazole gives a

TABLE 9. RESPONSE OF ORGANIC REFERENCE COMPOUNDS TO SODIUM FUSION AND FERROX TESTS

			Fusion results[a]				
Compound	RGN(s)	Heteroatoms present	N	S	P	X	Ferrox results[b]
Trinitrobenzoic acid	3,27,102	N, O	+	-	-	-	-
Picric acid	27,31,102	N, O	+	-	-	-	-
Dipicrylamine	7,27,102	N, O	+	-	-	-	-
Dimethylaminoazobenzene	7,8	N	+	-	-	-	-
Aminopropionitrile	7,26	N	+	-	-	-	+
Aminothiazole	7	N, S	[c]	+	-	-	+
Benzoyl peroxide	30,102	O	-	-	-	-	+
Malonic nitrile	3,26	N, O	+	-	-	-	+
Toluene diisocyanate	18,107	N, O	+	-	-	-	-
Malathion	13,32	S, P	-	+	+	-	+
Parathion	27,32	N, S, P	+	+	+	-	+
Ethyl zimate	12,24	N, S	+	+	-	-	+
Vinyl acetate	13,103	O	-	-	-	-	+
Bromoxynil	13,17,26	N, O, Br	+	-	-	+	+

[a] + = positive test response
 - = negative test response

(continued)

[b] Stabilized with catechol derivative

[c] Nitrogen detected as thiocyanate

TABLE 9. (continued)

Compound	RGN(s)	Heteroatoms present	Fusion results[a] N	S	P	X	Ferrox results[b]
Chlorophenyl isocyanate	17,18,107	N, O, Cl	+	-	-	+	-
Styrene	16,28,103	None	-	-	-	-	+
Ethylene glycol monomethyl ether	4,14	O	-	-	-	-	+
Polypropylene	29,101	None	-	-	-	-	-
Propylene oxide	34,103	O	-	-	-	-	+
Methyl methacrylate	13,103	O	-	-	-	-	+
Ethyl acrylate	13,103	O	-	-	-	-	+
Mercaptobenzothiazole	20	S, N	c	-	-	-	+
Lannate	9	N, O, S	+	+	-	-	+
Hydroxyacetophenone	19,31	O	-	-	-	-	+
Mercaptoethanol	4,20	O, S	-	+	-	-	+
n-Butyl acrylate	13,103	O	-	-	-	-	+
Acrolein	5,103	O	-	-	-	-	+
Diacetone alcohol	4,19	O	-	-	-	-	+
Diphenamide	6	N, O	+	-	-	-	+

[a] + = positive test response
- = negative test response

(continued)

[b] Stabilized with catechol derivative

[c] Nitrogen detected as thiocyanate

TABLE 9. (concluded)

Compound	RGN(s)	Heteroatoms present	Fusion results[a] N	S	P	X	Ferrox results[b]
Hexene	28	None	-	-	-	-	-
Decane	28	None	-	-	-	-	-
TEPA	7,32	N, P, O	+	-	+	-	+
Acetamide	6	N, O	+	-	-	-	+
Naphthalene	16	None	-	-	-	-	-
Polysulfide polymer	14,20,105	S	-	+	-	-	+

[a] + = positive test response
- = negative test response

[b] Stabilized with catechol derivative

[c] Nitrogen detected as thiocyanate

positive response. However, for compounds containing oxygen as the only heteroatom (ethers, esters, etc.), the ferrox test gave consistently positive results. For hydrocarbons and for several chlorinated hydrocarbons tested (methylene chloride, chloroform), the responses were negative. Styrene, however, gave a positive result which was attributed to the catechol stabilizer added.

The ferrox test can therefore be used to distinguish between hydrocarbons or halogenated hydrocarbons and organic comounds containing oxygen. The test is inconclusive when applied to organic materials containing nitrogen or sulfur.

3.4.5 PS 5 -- Organic Functionality Tests

3.4.5.1 PS 5A -- Tests for Hydrocarbon Classification

Two tests were investigated for classification of hydrocarbons as aromatic, saturated, or unsaturated. The Friedel-Crafts test for aromatics is performed first in the sequence, followed by the Baeyer test for unsaturation.

Since only a limited number of hydrocarbons are present among the reference compounds selected for this study, some additional hydrocarbons were selected for verification of these test procedures. The results of these tests are presented in Table 10. Results correspond to those expected, and no anomalous results were observed. In all cases the test results were clear and easily interpreted.

3.4.5.2 PS 5B -- Tests for Organic Functional Groups Containing Oxygen

Eight functional group tests were performed on all of the organic reference compounds. These eight tests were as follows:

- pH test for organic acids (RGN 3) (conducted as part of PS 1)
- Vanadium oxinate test for alcohols (RGN 4)
- Dinitrophenylhydrazine (DNPH) test for carbonyl compounds (RGN's 5 or 19)
- Schiff's test for aldehydes (RGN 5)
- Hydroxamate test for esters (RGN 13)
- Iodine test for esters (RGN 14)
- Starch-iodide test for organic peroxides (RGN 30)
- Ferric chloride test for phenols (RGN 31)

TABLE 10. RESULTS OF HYDROCARBON CLASSIFICATION TESTS FOR A SERIES OF HYDROCARBONS

Compound	Results[a]	
	Friedel-Crafts test	Baeyer test
Stryene	+	+
Polypropylene	-	-
Hexene	-	+
Decene	-	+
Hexane	-	-
Decane	-	-
Naphthalene	+	-
Benzene	+	-
Hexadecane	-	-
Xylene (mixed isomers)	+	-
Octadiene	-	+
Toluene	+	-

[a] + = positive test response
- = negative test response

The results of the functional group tests for organic oxygen compounds are presented in Table 11. Ten false positive and one false negative test result were obtained in this series of tests (out of a total of 296 individual observations). Some specific comments regarding the test results are worthy of note.

With the exception of the iodine test for ethers all tests show good correlation with the expected test results. The high number of false positives (6 out of 37) in the iodine test for ethers is not surprising, since all compounds which can reduce iodine to iodide decolorize the reagent also. Among the compounds tested, dithiocarbamates, mercaptans, malathion, acrolein, and aminoproprionitrile gave false positive results. Therefore, positive test results should be disregarded when mercaptans, thiocarbamates, or aldehydes are found to be present.

Aside from the iodine test for ethers, the ferric chloride test for phenols is the only test which gave misleading results, one false positive and one false negative. First, a wide range of colors can be obtained depending on the phenolic compound involved, which increases the potential of misinterpretation. Aminoproprionitrile and mercaptoethanol reacted positive, whereas picric acid (trinitrophenol) did not. For the purpose of reactivity group identification false test results may be of limited significance when the test interferences are caused by substituents belonging to a reactivity group which has a higher incompatibility rating.

Although the vanadate test for alcohols led to the expected results it was difficult to observe a positive result. Many nonalcoholic compounds gave a yellow-orange or light brown-purple color which could be distinguished from the pink-orange characteristic color of the alcohol-vanadate complexes by using a positive control. The vanadate test for alcohols was preferred over the xanthate test because many of the nonalcohol compounds gave a similar color response to the xanthate test, as did alcohols. In addition, the procedure is lengthy and five different reagents are required.

No problems were encountered in distinguishing between positive and negative test responses in the dinitrodiphenylhydrazine tests for aldehydes and ketones. An alternate test for ketones and aldehydes, which uses azobenzenephenylhydrazide as a reagent was also tested with all compounds, but positive results -- a blue to pink chloroform layer -- were less distinct than results in the dinitrophenylhydrazine test which forms brightly colored precipitates with aldehydes and ketones. The Schiff's test for aldehydes gave equally satisfactory results. The p-nitrobenzene-diazonium tetrafluoroborate test for phenols and amines gives easily detectable results in the form of yellow, orange, or brown precipitates, but othro- and para-substituted aromatic amines and phenols do not react with the reagent (i.e., p-hydroxyacetophenone gave a negative test).

TABLE 11. SUMMARY OF FUNCTIONALITY TEST RESULTS FOR OXYGEN-CONTAINING ORGANIC COMPOUNDS

		Test results[a]							
Compound name	RGN(s)	RGN 3 pH	RGN 4 vanadium oxinate	RGN 5/19 DNPH	RGN 5 Schiff's test	RGN 13 hydroxamate	RGN 14 iodine	RGN 30 potassium-iodide starch	RGN 31 ferric chloride
Selenium diethyldithiocarbamate	12,24	-	-	-	-	-	+	-	-
Peracetic acid	3,30,104	+	-	-	-	-	-	-	+
Trinitrobenzoic acid	3,27,102	+	-	-	-	-	-	-	-
Picric acid	27,31,102	b	-	-	-	-	-	c	-
Dipicrylamine	7,27,102	-	-	-	-	-	-	-	-
Dimethylaminoazobenzene	7,8	-	-	-	-	-	+	-	-
Aminopropionitrile	7,26	-	-	-	-	-	b	-	-
Aminothiazole	7	-	-	-	-	-	-	b	-
Benzoyl peroxide	30,102	-	-	-	-	b	-	-	+
Malonic nitrile (cyanoacetic acid)	3,26	+	-	-	-	-	-	-	-
Toluene diisocyanate	18,107	-	-	-	-	-	-	-	-
Malathion	13,32	-	-	-	-	+	b	-	-
Parathion	27,32	-	-	-	-	-	-	-	-
Ethyl zimate	12,24	-	-	-	-	-	b	-	-
Vinyl acetate	13,103	-	-	-	-	+	-	-	-
Bromoxynil	13,17,26	-	-	-	-	+	-	-	-
Chlorophenyl isocyanate	17,18,107	-	-	-	-	-	-	-	-
Styrene	16,28,103	-	-	-	-	-	-	-	-
Ethylene glycol monomethyl ether	4,14	-	+	-	-	-	+	-	-
Polypropylene	29,101	-	-	-	-	-	-	-	-
Propylene oxide	34,103	-	-	-	-	-	-	-	-
Methyl methacrylate	13,103	-	-	-	-	+	-	-	-
Ethyl acrylate	13,103	-	-	-	-	+	-	-	-
Mercaptobenzothiazole	20	-	-	-	-	-	b	-	-
Lannate	9	-	-	-	-	-	-	-	-
Hydroxyacetophenone	19,31	-	-	+	-	-	-	+	-
Mercaptoethanol	4,20	-	+	-	-	-	b	-	-
N-butyl acrylate	13,103	-	-	-	-	+	-	-	-
Acrolein	5,103	-	-	+	+	-	b	-	-
Diacetone alcohol	4,19	-	+	+	-	-	-	-	-
Polysulfide polymer	14,20,105	b	-	-	-	-	+	-	-
Hexene	28	-	-	-	-	-	-	-	-
Decene	28	-	-	-	-	-	-	-	-
TEPA	7,32	-	-	-	-	-	-	-	-
Acetamide	6	-	-	-	-	-	-	-	-
Naphthalene	16	-	-	-	-	-	-	-	-
Diphenamide	6	-	-	-	-	-	-	-	-

[a] + = positive test response
- = negative test response

[b] False positive

[c] False negative

3.4.5.3 PS 5C -- Tests for Organic Functional Groups Containing Nitrogen

Six functional group tests were conducted for organic functional groups containing nitrogen. These tests were performed only for those reference compounds containing nitrogen, sulfur, or phosphorus, since these compounds most realistically represent those materials upon which these test procedures will be applied according to the test schemes. The test procedures conducted are as follows:

- Oxamide test for amides and nitriles (RGN's 6 and 26)
- p-Nitrobenzene-diazonium tetrafluoroborate (NBDTF) test for amines (RGN 7)
- Dimethylaminobenzaldehyde (DMAB) test for azo compounds (RGN 8)
- Glutaconic aldehyde test for hydrazines (RGN 8)
- Diphenylcarbohydrazide (DPCH) test for carbamates (RGN 9)
- Tetrabase fusion test for organic nitro compounds (RGN 27)

The results of the functionality tests for organic nitrogen compounds (as well as organic sulfur compounds) are compiled in Table 12. A total of 166 observations were made; four false positive results were recorded. In addition, in a number of cases, color development was observed before the completion of the test procedure, necessitating the inclusion of controls into the test procedures. In two cases, the intense color of the reference compounds interfered with the test results themselves.

The oxamide test for amides and nitriles gave consistent results, but involves two heating steps. The test requires caution to avoid charring of the test material during heating. This procedure does not distinguish between amides and nitriles, but these two classes of compounds have very similar compatibility characteristics so it was not considered critical to distinguish between these two classes.

Tests for azo compounds and hydrazines were found to be very sensitive and easy to perform and no particular difficulties were encountered. Likewise the tetrabase fusion test for nitro compounds was found to be easy to perform and provided reasonably clear results. The diphenylcarbohydrazide test for carbamates was found to give false positive results, especially with the isocyanates (i.e., isocyanates are undoubtedly converted to carbamates during the course of the test procedure).

The dinitrochlorobenzene test for amines gave acceptable results with the reference compounds but is expected to provide marginal results for real waste materials. The test depends upon development of a brown ring which was found to be difficult to observe even for the pure reference compounds. For real waste materials, which are expected to be gross mixtures in many cases, the color of the test may be obscured by the waste material itself. A second

TABLE 12. SUMMARY OF FUNCTIONALITY TEST RESULTS FOR NITROGEN- AND SULFUR-CONTAINING ORGANIC COMPOUNDS

Compound	RGN(s)	Test results[a]							
		Nitrogen-containing RGN's						Sulfur-containing RGN's	
		RGN 6 or 26 oxamide	RGN 7 DNCB	RGN 8 DMAB	RGN 8 glutaconic aldehyde	RGN 9 DPCH	RGN 27 tetrabase	RGN 12 cupric chloride	RGN 20 lead acetate
Selenium diethyldithiocarbamate	12,24	-	-	-	-	d	-	+	-
Trinitrobenzoic acid	3,27,102	-	b	c	c	-	+	-	-
Picric acid	27,31,102	-	-	c	c	-	+	-	-
Dipicrylamine	7,27,102	-	+	-	-	-	+	-	-
Dimethylaminoazobenzene	7	b	+	c	c	-	b	-	-
Aminopropionitrile	7,26	+	+	-	-	-	-	-	-
Aminothiazole	7,8	-	+	-	-	-	-	-	-
Malonic nitrile (cyanoacetic acid)	3,26	+	-	-	-	-	-	-	-
Toluene diisocyanate	18,107	-	-	-	-	d	-	-	-
Malathion	13,32	-	-	-	-	-	-	-	-
Parathion	27,32	-	-	-	-	-	+	-	-
Ethyl zimate	12,24	-	-	-	-	-	-	+	-
Bromoxynil	13,17,26	-	-	-	-	-	-	-	-
Chlorophenyl isocyanate	17,18,107	-	-	-	-	d	-	-	-
Mercaptobenzothiazole	20	-	d	-	-	-	-	-	+
Lannate	9	+	-	-	-	+	-	-	+
Mercaptoethanol	4,20	-	-	-	-	-	-	-	+
Polysulfide polymer	14,20,105								
TEPA	7,32	-	-	-	-	-	-	-	-
Acetamide	6	+	-	-	-	-	-	-	-
Diphenamide	6	+	-	-	-	-	-	-	-
Phenylhydrazine	8	-	-	-	+	-	-	-	-

[a] + = positive test response
- = negative test response

[b] Compound interferes because it is intensely colored

[c] See a color change without one of the test steps; interference can be identified with a control

[d] False positive

test for amines, the NBDTFB gave more definitive results but also gives positive results with phenols. Results for both tests are presented in Table 12, and reagents for both are included in the test kit. Amines can be distinguished from phenols by means of the ferric chloride test for phenols.

3.4.5.4 PS 5D -- Tests for Organic Functional Groups Containing Sulfur

Two tests were conducted for organic functional groups containing sulfur. These two tests were applied to the organic reference compounds containing sulfur and/or nitrogen. The test procedures are:

- The cupric chloride test for dithiocarbamates (RGN 12)
- Lead acetate test for organic sulfides

The test results are summarized in Table 12. No anomalous results were obtained, and test responses were easily observed and interpreted.

3.4.6 PS 6 -- Inorganic Functionality Tests

Several metal-detecting reagents were tested for their suitability for the purpose of reactivity group determination. Although the reactions with dimethylbenzylidenerhodanine, diphenylcarbazone, and quinalizarine were easy to observe, the reactions are not general enough for the purpose of classification of heavy metals, alkali, and alkaline earth metals in one group each.

Zinc sulfide and ammonium sulfide were used to detect heavy metals. The test is simple and observations are easy with no interferences if test conditions are observed: acidic with zinc sulfide and almost neutral (a pH of about 5) for ammonium sulfide.

Elemental metals were easily identified with phosphomolybdic acid. No interferences were encountered. Alkali and alkaline earth metals were detected by their reactivity with water. Those alkali or alkaline earth metals that are not water reactive (e.g., magnesium) are classified as other metals by this scheme. Inorganic fluorides were determined with a mixture of zirconium nitrate and sodium alizarinate. The decolorization of the reagent by fluoride was easily observed in a spotplate. The observations were equally good by spotting the test solution of zirconium-alizarin impregnated filter paper, but some additional time is required to prepare the paper which can only be stored for a limited time without fading. The test results are summarized in Table 13.

3.4.7 Summary of Functionality Test Results

Specific comments regarding the test procedures and results are contained in Sections 3.4.1 through 3.4.6. In particular, information concerning functionality tests is discussed in Sections 3.4.5 through 3.4.6. In all, over 750 individual functionality test observations were made (PS 4, 5, and 6). Sixteen false positive results and six false negative results

TABLE 13. SUMMARY OF TEST RESULTS FOR INORGANIC COMPOUNDS AND ELEMENTAL METALS

		RGN 15	RGN 22,23,24	RGN 24				
Compound	RGN(s)	Zirconium alizarinate	Phosphomolybdic acid	Quinalizarine	Dimethylbenzyl-idenerhodanine	Zinc sulfide	Ammonium sulfide	Diphenyl-carbazone
Cadmium carbonate	24	-	-	-	-	-	+	+
Ferrous sulfate	(105)	-	-	-	-	-	+	-
Silver sulfate	24	-	-	-	+	+	+	-
Cuprous chloride	24,(105)	-	+	+	+	+	+	+
Cupric chloride	24	-	-	+	+	+	+	+
Nickel nitrate	24	-	-	+	-	+	+	+
Mercuric chloride	24	-	-	-	+	+	+	+
Cobalt chloride	24	-	-	+	-	-	+	-
Zinc acetate	24	-	-	+	-	-	-	-
Lead acetate	24	-	-	-	-	+	+	+
Barium nitrate	24	-	-	+	-	-	-	-
Antimony potassium tartrate	24	-	-	-	-	-	+	-
Stannous chloride	24/105	-	+	-	-	-	+	+
Manganese sulfate	24	-	-	+	-	-	-	-
Magnesium nitrate	24	-	-	-	-	-	-	-
Potassium platinic acid	24	-	-	-	-	+	+	-
Aluminum nitrate	24	-	-	-	-	-	-	-
Chromium trioxide	24,102,104	-	-	-	-	-	+	+
Cadmium	22,24	-	+	+	-	-	+	-
Sodium	21,10	-	-	-	-	-	-	-
Arsenic pentasulfide	24,33	-	-	-	-	-	-	-
Chromium	22,24	-	-	-	-	-	-	-
Copper	22	-	+	-	-	-	+	+
Lead	22,24	-	+	-	-	-	+	-
Nickel	22,24	-	+	-	-	-	+	-
Barium	21	-	+	+	-	-	-	+
Barium iodide	24	-	-	+	-	-	-	+
Barium oxide	10,24	-	-	+	-	-	-	+
Calcium hypochloride	10,104	-	-	-	-	-	-	-
Selenium diethyldithiocarbamate	12,24	-	-	-	-	-	-	-
Hydrofluorosilic acid	1,15	-	-	-	-	-	-	-
Fluoboric acid	2,15	-	-	-	-	-	-	-
Hexafluorophosphoric acid	1,15	-	-	-	-	-	-	-
Sodium fluoride	15	-	-	-	-	-	-	-
Zinc	23	-	+	-	-	-	-	-
Tin	23	-	+	-	-	-	-	-
Aluminum	22	-	-	-	-	-	-	-
Potassium cyanide	11	-	-	-	-	-	-	-

[a]+ = positive test response
- = negative test response

were recorded. A summary of the reliability of the test procedures employed as applied to the reference compounds is shown in Table 14. In light of the multiple functionality of many of the reference compounds, the frequency of false positive and false negative observations is considered to be acceptable.

3.5 RESULTS OF BLIND ANALYSES USING THE TEST SCHEMES

3.5.1 Results of Blind Analyses of the Reference Compounds

The test schemes were evaluated by subjecting the reference compounds to the schemes. The reference compounds were submitted as unknowns after concealing their identities and assigning numbers to them. Testing was carried out following the sequence indicated in the schemes and assigning RGN's based on the individual test results.

The experimentally determined RGN's for each compound are summarized in Table 15 for the purpose of comparison with their known RGN's. Several of these compounds exhibited properties during testing which were inconsistent with their assigned RGN's (Appendix 1 of Reference 1). After evaluation of the chemical structure of these compounds, it was discovered that some of the RGN's were incorrectly assigned, and these were corrected. The RGN changes are indicated in Table 15.

Two blind test runs (A and B) were conducted by two different technicians to determine the reproducibility of the results. The second set of blind analyses was carried out by a technician who was not involved in the development of the test procedures and had no specific training in chemical analysis. He was given the test kit instruction manual and also a draft of this report. He was instructed in how to use the test equipment and given minimal instructions on the critical points of each test, e.g., interpretation of the flame test in terms of flammability, explosiveness, combustibility, and residue. Also some initial assistance was given in interpretation of the precipitate formation or color development for certain tests. Aside from this initial assistance, he carried all reference compounds through the test scheme and assigned RGN's without further assistance. The test results for the technician new to the program are presented in column B of Tables 15, 16, and 17.

Potassium cyanide and dimethylaminazobenzene were not submitted to the blind tests to prevent unnecessary exposure as recommended by the Acurex safety officer. Several other compounds were also eliminated because sufficient quantities for testing were not available, as indicated in Table 15. Of the remaining reference compounds (test run A), seven were assigned incorrect RGN's or no RGN could be determined. For three of these compounds the results are explained by their chemical behavior during testing. Chromium was not detected as elemental metal because of its extreme insolubility under the test conditions. Barium iodide, barium oxide, and barium cannot be detected as barium sulfide, and toluene diisocyanate showed positive amine and ester test results, while the isocyanate group was not detected because no qualitative test method was available. Other

TABLE 14. GENERAL SUMMARY OF FUNCTIONALITY TEST RESULTS

Test name	Parameter	Tests conducted	Positive results expected	False positive results	False negative results
Sodium fusion					
Cyanide/thiocyanate	Organic nitrogen compounds	40	18	0	0
Sulfide	Organic sulfur compounds	40	8	0	0
Phosphate	Organic phosphorus compounds	40	2	0	0
Halide	Organic halogen compounds	40	2	0	0
Ferrox test	Organic oxygen compounds	40	10	1	1
pH	Acids (organic)	40	3	2	0
Vanadium oxinate	Alcohols	40	3	0	0
Dinitrophenylhydrazine	Aldehydes and ketones	40	3	0	0
Schiff's test	Aldehydes	40	1	0	0
Hydroxamate	Esters	40	6	1	0
Iodine test	Ethers	40	3	6	0
Ferric chloride	Phenols	40	2	1	1
Potassium iodide-starch	Peroxides	40	2	0	0
Oxamide	Amides and/or nitriles	22	5	0	0
Dinitrochlorobenzene	Amines	22	4	1	0
Dimethylaminobenzaldehyde	Azo compounds	22	1	0	0
Glutaconic aldehyde	Hydrazines	23	1	0	0
Diphenylcarbohydrazide	Carbamates	22	1	3	0
Tetrabase fusion	Nitro compounds	22	4	0	0
Copper chloride	Dithiocarbamates	22	2	0	0
Lead acetate	Mercaptans and other organic sulfides	22	3	0	0
Baeyer's test	Unsaturated hydrocarbons	13	4	0	0
Friedel-Crafts test	Aromatic hydrocarbons	3	3	0	0
Zirconium alizarinate	Inorganic fluorides	10	5	0	0
Phosphomolybdic acid	Elemental metals	16	10	0	0
Sulfide	Heavy metal compounds	16	16	0	0
Total		755	122	15	2

TABLE 15. RESULTS OF DUPLICATE BLIND ANALYSES OF THE REFERENCE COMPOUNDS USING THE TEST SCHEMES

Compound	RGN(s)	RGN(s) (experimental) Run A	RGN(s) (experimental) Run B	Comments
Chromium oxide	2[a],24,104[a]	24,102	104,2	Reacts like explosive, color interference
Cadmium	22,24	22,24,102	22,24	Reacts like explosive
Sodium	10[f],21,102,105,107	10,102,107	--	Reacts like explosive
Arsenic pentasulfide	24[a],33	33	--	
Chromium	23,24	ND	ND	Some acid reaction, insoluble under test conditions
Copper	23,24	23,24	23,24	
Lead	23,24	23,24	23,24	
Nickel	22,24	22,24	22,24	
Barium	10,21,24[a],107	10,21,102,107	10,21,24,102,107	Reacts like explosive
Barium iodide	24[a]	ND	104	
Barium oxide	10,24[a],107[a]	10	10,22	
Calcium hypochlorite	10[f],104	10,104	10,104	
Selenium diethyl-dithiocarbamate	12,24	12	12,19	
Hydrofluorosilicic acid	1,15	1,15	1,15	
Peracetic acid	3,30[b],104[f]	3,104	3	Eliminated from scheme after PS 1
Fluoboric acid	2[f],15	2,15	2,15	
Hexafluorophosphoric acid	2[f],15	2,15	2,15	
Hydrofluoric acid	1,15	1,15	1,15	
Trinitrobenzoic acid	3[f],27,102	3	3,6/26[c]	Eliminated from scheme after PS 1
Picric acid	3,27[b],31[b],102	3,102	3,27,31	Eliminated from scheme after PS 3
Dipicrylamine	7,27,102	102	3[c],31[c]102	
Aminoproprionitrile	7,26	6/26,7,14[c]	6/26,7	
Aminobenzothiazole	7	7	7,6/26[c]	
Benzoyl peroxide	30,102	13[c],30,102	30,104,102	
Cyanoacetic acid	3,26	3,6/26	3	
Toluene diisocyanate	18[d],107[a]	7[c],13[c]	9	May hydrolyze during test
Hydroxylamine·HCl	105	ND		

(continued)

ND: No RGN determined

-- Not done, insufficient sample

6/26: Same test for either RGN

[a] Not detectable by test used

[b] Not tested for this RGN due to elimination from scheme

[c] Incorrect RGN

[d] No test available for this RGN

[e] RGN not detected

[f] Corrected RGN

TABLE 15. (concluded)

Compound	RGN(s)	RGN(s) (experimental) Run A	RGN(s) (experimental) Run B	Comments
Malathion	13^{f},32	3,13,32	32	pH 3
Parathion	$27^{e,f}$,32	4^{c},32	27,32,$6/26^{c}$	
Ethyl zimate	12,24	12,14^{c}	12,14	
Vinyl acetate	13,103^{c}	13	13	
Bromoxynil	$13^{e,f}$,17,26^{e}	17	13,17,26,107^{a}	
p-Chlorophenylisocyanate	17,18^{d},107^{a}	17	9^{c},17,$6/26^{c}$	
Styrene	16,28,103^{d}	16,28	16,28	
Ethylene glycol-monomethylether	4,14^{e}	4	4,14	
Polypropylene	29,101^{a}	29	--	
Propyleneoxide	34^{d},103^{d},107	4,107	107	Hydrolyzes to alcohol
Methyl methacrylate	13,103^{d}	4^{c},13	13	
Ethyl acrylate	13,103^{d}	13	13	
Mercaptobenzothiazole	20	7^{c},20	20	
Lannate	9	9	9	
Hydroxyacetophenone	19,31	19,31	19	
Mercaptoethanol	4^{e},20,105^{f}	20,105	20	
b-Butylacrylate	13,103^{d}	ND	13	
Acrolein	5,103^{d}	5	5	
Diacetone alcohol	4^{e},19	19	4,19	
Aldicarb	9	ND	17^{c},32^{c}	
Polysulfide	$14^{e,f}$20,101^{a},$105^{e,f}$	3,20	20,14	
Hexene	28	28	28	
Decene	28	28	28	
Tepa	7^{f},32	32	--	
Acetamide	6	6/26	6/26	
Naphthalene	16	16	16	
Diphenamide	6	ND	6/26	
n-Decane	29	29	29	

ND: No RGN determined

-- Not done, insufficient sample

6/26: Same test for either RGN

[a] Not detectable by test used

[b] Not tested for this RGN due to elimination from scheme

[c] Incorrect RGN

[d] No test available for this RGN

[e] RGN not detected

[f] Corrected RGN

TABLE 16. BLIND TEST RESULTS FOR OXYGEN FUNCTIONAL GROUPS[a]

Compound	RGN(s)	RGN 4 vanadate		RGN 5 or 19 DNPH		RGN 5, Schiffs test		RGN 13 hydroxamate		RGN 14 iodine		RGN 31 ferric chloride	
		A	B	A	B	A	B	A	B	A	B	A	B
Selenium diethyl-dithiocarbamate	12,24	-	-	-	++	-	-	-	-	-	-	-	-
Trinitrobenzoic acid	3,27,102	b	-	b	-	b	-	b	-	b	-	b	-
Picric acid	3,27,31,102	b	-	b	-	b	-	b	-	b	-	b	+
Dipicrylamine	7,27,102	b	-	b	-	b	-	b	-	b	-	b	-
Aminoproprionitrile	7,26	-	++	-	-	-	-	-	-	-	-	-	-
Aminobenzothiazole	7	-	-	-	-	-	-	-	-	-	-	-	-
Benzoyl peroxide	30,102,(13)	-	b	-	b	-	b	+	b	-	b	-	b
Cyanoacetic acid	3,26	-	-	-	-	-	-	-	-	-	-	-	-
Toluene diisocyanate	18,107	-	-	-	-	-	-	++	-	-	-	-	-
Malathion	13,32	-	c	-	c	-	c	+	c	-	c	-	c
Parathion	27,32	-	c	-	c	-	c	--	c	-	c	-	c
Ethyl zimate	12,24	-	-	-	-	-	-	-	-	++	++	-	-
Vinyl acetate	13,103	-	-	-	-	-	-	+	+	-	-	-	-
Bromoxynil	13,17,26	-	-	-	-	-	-	--	+	-	-	-	-
Chlorophenyl isocyanate	17,18,107	-	-	-	-	-	-	-	-	-	-	-	-
Ethylene glycol monomethyl ether	4,14	+	+	-	-	-	-	-	-	--	+	-	-
Propylene oxide	29,101	+	b	-	b	-	b	-	b	-	b	-	b
Methyl methacrylate	34,103,107	++	-	-	-	-	-	+	+	-	-	-	-
Ethyl acrylate	13,103	-	-	-	-	-	-	+	+	-	-	-	-
Mercaptobenzothiazole	20	-	-	-	-	-	-	-	-	-	-	-	-
Lannate	9	-	-	-	-	-	-	-	-	-	-	-	-
Hydroxyacetophenone	19,31	--	--	+	+	+	-	-	-	-	-	+	--
Mercaptoethanol	4,20,105	--	--	-	-	-	-	-	-	-	-	-	-
n-Butyl acrylate	13,103	-	-	-	-	-	-	--	+	-	-	-	-
Acrolein	5,103	-	-	+	+	+	+	-	-	-	-	-	-
Diacetone alcohol	4,19	--	+	+	+	-	-	-	-	-	-	-	-
Aldicarb	9	-	c	-	c	-	c	-	c	-	c	-	c
Polysulfide	20,14,101,105	-	-	-	-	-	-	-	-	--	+	-	-
Tepa	7,32	-	c	-	c	-	c	-	c	-	c	-	c
Acetamide	6	-	-	-	-	-	-	-	-	-	-	-	-
Diphenamide	6	-	-	-	-	-	-	-	-	-	-	-	-
False positives ++		1	1	0	1	0	0	1	0	1	1	0	0
False negatives --		3	2	0	0	0	0	3	0	2	0	0	1
Total number of tests		28	25	28	25	28	25	28	25	28	25	28	25

[a] + = positive test response
- = negative test response

[b] Functional tests not done, compound eliminated earlier in testing sequence

[c] Insufficient test material remaining for testing

TABLE 17. BLIND TEST RESULTS FOR NITROGEN AND SULFUR FUNCTIONAL GROUPS[a]

Compound	RGN(s)	RGN 9 DPCH		RGN 8 DMAB		RGN 8 glutatonic acid		RGN 6 or 26 oxamide		RGN 7 NBDTFB		RGN 3/31 ferric chloride		RGN 27 tetra-base		RGN 12 cupric chloride		RGN 20 lead acetate	
		A	B	A	B	A	B	A	B	A	B	A	B	A	B	A	B	A	B
Selenium diethyl-dithio carbamate	12,24	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-
Trinitrobenzoic acid	3,27,102	b	-	b	-	b	-	b	++	b	-	b	-	b	-	d	d	d	d
Picric acid	3,27,31,102	b	-	b	-	b	-	b	-	b	-	b	-	b	+	d	d	d	d
Dipicrylamine	7,27,102	b	-	b	-	b	-	b	-	b	--	b	-	b	--	d	d	d	d
Aminoproprionitrile	7,26	-	-	-	-	-	-	+	+	+	+	-	-	-	-	d	d	d	d
Aminobenzothiazole	7	-	-	-	-	-	-	-	++	+	+	-	-	-	-	-	-	-	-
Cyanoacetic acid	3,26	-	-	-	-	-	-	+	--	-	-	-	-	-	-	d	d	d	d
Toluene diisocyanate	18,107	-	++	-	-	-	-	-	-	++	-	-	-	-	-	d	d	d	d
Malathion	13,32	-	c	-	c	-	c	-	c	-	c	-	c	-	c	-	-	-	-
Parathion	27,32	-	-	-	-	-	-	-	++	-	-	-	-	--	+	-	-	-	-
Ethyl zimate	12,24	-	-	-	-	-	-	-	-	-	-	-	-	-	-	+	+	-	-
Bromoxynil	13,17,26	-	-	-	-	-	-	--	+	-	-	-	-	-	-	d	d	d	d
Chlorophenyl isocyanate	17,18,107	-	++	-	-	-	-	-	++	-	-	-	-	-	-	d	d	d	d
Mercaptobenzothiazole	20	-	-	-	-	-	-	-	-	++	-	-	-	-	-	-	-	+	+
Lannate	9	+	+	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Mercaptoethanol	4,20,105																	+	+
Aldicarb	9	--	c	-	c	-	c	-	c	-	c	-	c	-	c	-	c	-	c
Polysulfide	20,14,101,105															--	+	+	+
Tepa	7,32	-	c	-	c	-	c	-	c	--	c	-	c	-	c	d	d	c	d
Acetamide	6	-	-	-	-	-	-	+	+	-	-	-	-	-	-	-	-	-	-
Diphenamide	6	-	-	-	-	-	-	--	+	-	-	-	-	-	-	-	-	-	-
Total		16	16	16	16	16	16	16	16	16	16	16	16	16	16	11	10	11	10
False positives ++		0	2	0	0	0	0	0	4	2	0	0	0	0	0	0	0	0	0
False negatives --		1	0	0	0	0	0	2	1	1	1	0	0	1	1	0	0	0	0

[a] + = positive test response
 - = negative test response

[b] Functional group test not done, compound eliminated earlier in testing sequence

[c] Insufficient test material remaining for testing

[d] Testing not required (according to results of PS 4)

difficulties were the detection of water reactivity (RGN 107) which was defined as reaction with visible gas evolution or spattering. This resulted in nondetection of RGN 107 for three compounds: barium oxide, toluene isocyanate, and chlorophenyl isocyanate. In addition, polymerizable compounds were not detectable. Several materials (chromium trioxide, cadmium, and barium) reacted violently during the flame test, although they are not considered explosives. The functional group results for the blind analyses are presented in Tables 16 and 17.

The comparison of results from both test replicates show good correlation of most RGN's. The individual functionality tests also showed good agreement, with 18 disagreeing individual test results out of the 261 individual determinations.

3.5.2 Results of Blind Analyses of Other Pure Compounds

Two sets of unknown laboratory chemicals were tested by following the test scheme. Six unknowns were mixtures of two compounds (HWC-X, 1-6); twelve unknowns were single compounds (HWC-X, 11-22). The RGN's determined from testing as compared to their actual RGN's are presented in Table 18. Out of the 12 single unknown compounds tested all RGN's were correctly assigned for 9 compounds and for the remaining 3 the most significant RGN's were assigned. Four RGN's were incorrectly assigned (false positive) and one RGN was not detected. For the six unknown mixtures, the RGN's were only partially identified because several of the tests which would identify functional groups of the second component of the mixture were not conducted according to the test scheme in the following instances: (1) in the presence of certain functional groups, (2) at a specific pH (less than 3,) or (3) where the mixture exhibited explosive behavior during the flame test. Nevertheless, the most significant RGN's were assigned for all of the six mixtures.

3.6 DEVICE FOR THE DETERMINATION OF THE COMPATIBILITY OF UNKNOWN MATERIALS

Two important criteria for evaluating the compatibility of two or more substances when mixed together are heat generation and gas evolution. Both can create hazards in the form of pressure increases and formation of toxic gases. These effects are measured with a simple mixing device which is easy to assemble and use under field conditions. A diagram of the device is shown in Figure 8.

The device consists of a 120-mL glass reaction vessel equipped with a rubber stopper with holes for a hand-operated glass stirring rod with a Teflon paddle, a thermocouple (K-type stainless steel, Omega), a burette for adding liquids, and Teflon tubing which is connected to a 50-mL impinger. The temperature is measured with a battery-operated digital thermometer (Omega Model 727C). The reaction vessel and the impinger bottle are held in a wooden block; the cavity for the reaction vessel is deep enough to provide protection in the event of glass breakage but still permits visual observation of the reaction. The reaction vessel is held by a clamp and metal stand. Violent reactions during mixing can be detected by adding small

TABLE 18. RESULTS OF BLIND ANALYSES OF SELECTED PURE COMPOUNDS AND BINARY MIXTURES

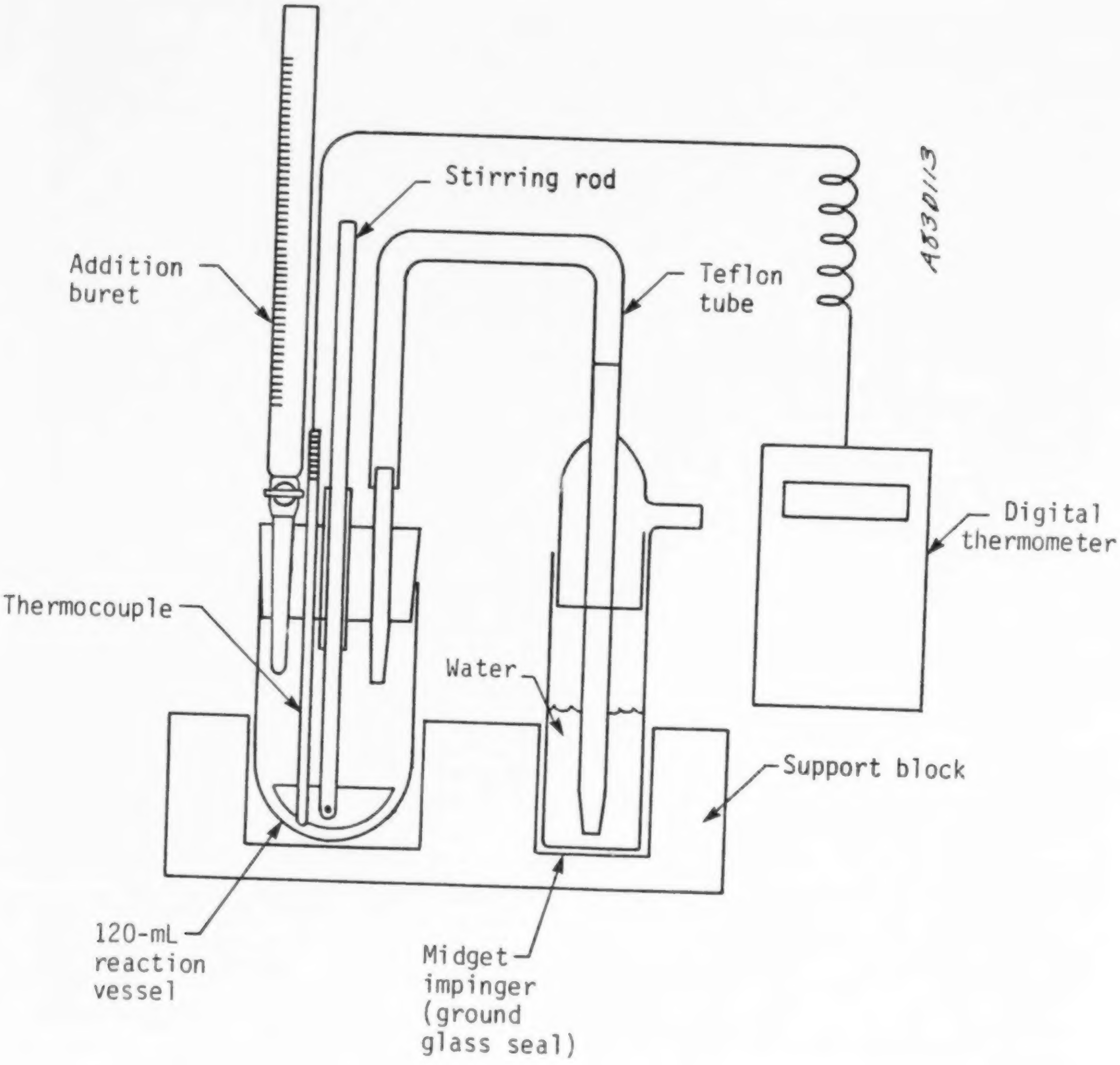
Hazardous waste combination	Compound	RGN(s) (known)	RGN(s) (experimental)
Two Components			
1	Picric acid, ethylene glycol monomethyl ether	4[b],14[a],31,27[a],102	3,102
2	Mercaptobenzothiazole, diacetone alcohol	20,4[b],19,105	20,19,7,105
3	Styrene, hydroxyacetophenone	16[a],103,4,19,31,28[d]	4,19,31,32[c],16,28
4	Bromoxynil, vinyl acetate	17,26[b],13,103[d]	9,17,13
5	Mercaptoethanol, hexane	4,20,28[a]	4, 20
6	Chlorophenyl, isocyanate	17,18[d],107[b]	17,19[c],6/26[c]
Single Compounds			
11	Iodine	104,2	104,2
12	Sodium borohydride	105	105,14[c],102,17[c]
13	Sodium diethyldithiocarbamate	12	12
14	Stannous sulfate	24,105,1	1,24,105
15	Benzoic acid	3	3,19[c]
16	Bromobenzene	17	17
17	Phenylhydrazine	8	14[c],8
18	O-Nitroaniline	7,27[b]	7
19	Pyrogallol	3,31,105	3,31,105
20	Tin (granules)	22,24	22,24
21	Biphenyl	16	16
22	Hydrofluoric acid	1,15,106	1,15,106

[a] Not tested for this RGN due to elimination from test scheme

[b] RGN not detected

[c] Incorrect RGN

[d] No test available for this RGN



Stirring rod
Addition buret
Teflon tube
A83D113
Digital thermometer
Thermocouple
Water
Support block
120-mL reaction vessel
Midget impinger (ground glass seal)

Figure 8. Device for determining the effects of mixing of unknown materials.

amounts of the test materials to a test tube before carrying out the reaction in the mixing device. If the mixture creates heat or forms gases or fumes in test tube quantities no mixing with larger quantities is necessary. To avoid any accidents, the experiment should be carried out behind a safety shield.

Pressure changes are detected by observing bubbles in the impinger. The pressure changes which can be detected with the device used in the present experiment are sufficiently small to aid in determining noncompatibility for instantaneous reactions. Very small pressure increases or pressure buildup over long periods of time are not detected with this device.

The reactivity tests are carried out by placing 3 to 5g of the solid or 3 to 5 mL of the liquid test material in the reaction vessel, then closing the stoppers and adjusting the thermocouple so it reaches the test material. The impinger is filled with 10 mL of water and connected to the reaction vessel. The temperature of the test material is recorded before the second (liquid) test material is added from a buret, the stopcock is closed, and both materials are mixed by turning the stirring rod. The reaction is observed for foaming, color changes, fumes, and temperature changes. Gas evolution is observed as bubbles appearing in the impinger solution. The gases can be identified by analysis of the impinger solution for sulfide, cyanide, carbonate, halogen, and acidity (pH). A series of compounds was selected for testing based on RGN's predicted to be incompatible from the hazardous waste compatibility chart. The test results are summarized in Table 19.

For the most part, those reactions that would be predicted (from basic chemical principles) to generate heat or gas were observed to do so in the test mixing device. In several cases, the compatibility chart predicts reactions that may not actually occur spontaneously. For example, the compatibility chart predicts heat evolved when mineral acids (RGN 2) and chlorinated organics (RGN 17) are mixed. No observable effect was noted when sulfuric acid was mixed with freon, however. This apparent anomaly is not considered to be a shortcoming of the mixing device.

Additional tests were conducted to collect and analyze gases formed during mixing. The gases were collected in a solution of 5 mL of 0.1N barium nitrate and 5 mL of 0.1N sodium hydroxide, and were analyzed for sulfide, cyanide, halogen, carbonate, and pH changes. The results are summarized in Table 20. The starting temperature (in the reaction vessel) and pH are listed as T_1 and pH_1, respectively. The final temperature and pH (in the impinger) are T_2 and pH_2.

Similar but more elaborate mixing devices have been used for determining the reactivity of waste materials with water (Reference 6 and 7). These reactors are pressure sealed and pressure changes are measured with pressure gauges. Toxic gases formed during the reaction with water such as HCN, H_2S, SO_2, CO, CO_2, NO_x are identified by connecting a gas detector manifold constructed from a series of Draeger tubes to the reaction vessel.

TABLE 19. RESULTS OF MIXING EXPERIMENTS

Compound I	Compound II	Predicted reaction[a]	ΔT (°C)	Gas evolution[b]	Comments
Barium oxide	Water	H	49	-	
Barium (metal)	Water	GF,H	82	+	
Propylene oxide	Water	--	3	+	
Trichlorotrifluoroethane	Sulfuric acid (3M)	H,GT	0	-	
Barium iodide	Hydrochloric acid (3M)	S	-1	-	Dissolves
Ammonium chloride	Sodium hydroxide (3M)	H	-12	-	
Sodium nitrite	Nitric acid (conc.)	H,GT	-10	+	
Acetamide	Nitric acid (conc.)	H,GT	-12	-	
Chlorophenyl isocyanate	Hydrochloric acid (2M)	H,G	-1	-	
Chlorophenyl isocyanate	Sodium hydroxide (5M)	H,F,G	17	+	
Phthalic acid	Sodium hydroxide (2.5M)	H	13	-	
Propylene oxide	Sodium hydroxide (5M)	H,P	0	-	
Glycerol	Sulfuric acid (1M)	H	5	-	
o-Nitroaniline	Sodium hydroxide (2.5M)	H	1	-	
Acrolein	Sodium hydroxide (2.5M)	H,P	48	+	Violent polymerization
Octadiene	Sulfuric acid (2M)	H	1		
Octadiene	Hydrochloric acid (2M)	H	3		
Benzoic acid	Zinc + water	GF	0	-	
Benzoic acid	Chromium + water	GF	2	-	
Picric acid	Zinc + water	GF	1	-	Dark green color formed
Phthalic acid	Nickel	GF	1	-	
Benzoyl peroxide	Methenamine	H,GT	4	-	
Octadiene	Nickel	H,E	2	-	
Propylene oxide	Diphenylamine	H,P	-5	+	
Propylene oxide	Sodium diethyl dithiocarbamate	U	2	+	

[a]Reactivity Code	Consequences
H	Heat generation
F	Fire
G	Innocuous and nonflammable gas generation
GT	Toxic gas generation
GF	Flammable gas generation
E	Explosion
P	Violent polymerization
S	Solubilization of toxic substances
U	May be hazardous but unknown

[b] + = positive test reponse
- = negative test response

TABLE 20. RESULTS OF THE ANALYSIS FOR EVOLVED GASES IN MIXING EXPERIMENTS

Compound	Reagent	T_1 (°C)	T_2 (°C)	Gas	pH_1	pH_2	Analysis
0.4g KCN in 20 mL H_2O	5 mL 2N H_2SO_4	17	19	Yes	14	13	CN^- not detected
2g Al powder	5 mL 2.5N NaOH	17	98	Yes	14	5	--
1g $Ca(OCl)_2$	6 mL 2N HCl	17	25	Yes	14	--	Cl^- as AgCl
0.4g Na_2S in 20 mL H_2O	5 mL 2N H_2SO_4	16	19	Yes	14	14	$S^=$, as PbS
0.4g $CaCO_3$ in 20 mL H_2O	5 mL 2N H_2SO_4	16	18	Yes	14	13	$CO_2^=$ as $BaCO_3$

3.7 FIELD EVALUATION OF THE TEST KIT

A field evaluation of the test kit was conducted on March 8 through 11, 1983 at the EPA Combustion Research Facility in Jefferson, Arkansas. Twenty-five hazardous waste samples were collected from two sites prior to the field evaluation by Mr. Richard Carnes, EPA Technical Advisor for the project. Four of the samples had two distinct layers. For these samples, the layers were sampled and analyzed separately, bringing the total number of samples analyzed to 29. The gross identity of the waste materials was known to the technical advisor but not to the field crew until after the testing was completed.

The tests were performed outside (day 1) on a temporary plywood working surface and in a trailer with a counter (days 2 and 3) because of inclement weather. The wind was gusting from 20 to 30 mph, and the temperature was in the mid-30's (°F). It is believed that these conditions simulated field conditions closely.

Only two significant difficulties were encountered in performing the tests under field conditions. The first was that several of the samples contained two phases and the kit was not equipped with a separatory funnel or equivalent device. Based on this experience, several small separatory funnels were added to the test kit equipment list. A second problem encountered was performing the tests under adverse weather conditions; a particular problem was wind. Under windy conditions reagent bottles and sample vials tipped over and the flame was difficult to maintain and control. Overall, the tests proved relatively easy to perform under field conditions.

A list of the samples including the composition and a summary of results is presented in Table 21. The detailed results of tests performed on the samples as well as the RGN's predicted from knowledge of the samples, are presented in Table 22. The results of PS 1 are given as N/N, A(4)/N, B(12)/R, etc. The first letter refers to the acidity or basicity. When the material is acidic or basic, the pH is given in parentheses. The second letter indicates the redox response of the material. R stands for reducing and O stands for oxidizing. The second column (PS 2) lists the response to the solubility/reactivity tests. The flame test results (PS 3) are given in the next column. Columns headed PS 4 through PS 6 present the results of the functionality tests. The first (PS 4) column gives the results of the sodium fusion and ferrox test for elements (N, X, S, P, O). An O in parentheses means oxygen is assumed to be present. The word "none" means that no heteroatoms were found, while -- indicates that the tests were not performed.

The last three columns list, respectively, the RGN's expected from the waste description, the RGN's found by the protocol, and additional special comments. A ?? in the predicted RGN's column indicates that insufficient information was available to identify potential RGN's.

Twenty-eight of the twenty-nine samples were categorized correctly (according to the impartial judgment of the project technical advisor onsite) in the field evaluation. Sample 6E was described as "waste solvents with amines" but was characterized as RGN's 10 (caustic), 24 (metal compounds), and 106 (water). Upon contacting the source of this sample it was found that indeed we had accidentally been given the water layer of a two-layer system. Amines dissolved in the water would account for the caustic (RGN 10) description and could act as a chelating agent to carry heavy metals into the water. Another sample, 3D, was described as ethanol wash (two layers). The upper layer was identified as aromatic hydrocarbon (RGN 16), but only water (RGN 106) was detected in the lower layer. Two oil samples containing PCB's (samples 16E and 17E) were identified only as aromatic hydrocarbons. The PCB levels in these samples were not known, but are assumed to be low (trace level) in which case the chloride was present at too low a level to be detected.

Sample 8DU was identified as organic but examination was not carried beyond the flame test. The flame test results indicated to a technician that the material might be an explosive. Further testing would have been at the discretion of a qualified supervisor. In fact, the material was the organic layer from the still bottom of a solvent recovery refinery. The lower (water) layer was found to contain metals. Thus, the flame behavior of 8DU was probably due to burning metallic materials in the sample.

Sample 3E, a waste naphtha, was found to have sulfur present but no sulfur compounds were identified. Sulfur is common in naphtha, either as elemental sulfur or as organic sulfides. Organic sulfur groups other than mercaptans, triocarbamates, and disulfides are not detected by the methods used in the test scheme. The failure to identify sulfur-containing organic species may also indicate a difference in sensitivity between the test for

TABLE 21. DESCRIPTION OF SAMPLES TESTED AND SUMMARY OF RESULTS OF THE FIELD EVALUATION

Sample[a]	Sample identification	Sample description	RGN(s)	
			Predicted	Found
1D	Dirty PCE	Yellow-brown liquid	17	4,17
2D	TCE still bottoms, oil, hydrocarbons	Dark brown emulsion very small upper layer	17	16,17
3DU	Ethanol wash and oils	Brown liquid (upper layer of two layers)	16,28	16
3DL	Ethanol wash and oils	Pale brown liquid (lower layer of two layers)	4,106	106
4DU	Toluene in thinner wash	Pale yellow liquid (upper layer of two layers)	16	16,19
4DL	Toluene in thinner wash	Pale brown liquid (lower layer of two layers)	106	106
5D	MEK, xylene, and solvents	Very light tan emulsion	16,19	4,16,19
6D	Acetone and MEK	Light purple emulsion	19	19
7D	MEK from paint manufacturing	Green-brown emulsion	19	16,19
8DU	Still bottoms from solvent recovery refinery	Dark brown slurry (upper layer of two layers)	b	102
8DL	Still bottoms from solvent recovery refinery	Pale yellow liquid (lower layer of two layers)	b	24,106
1E	Organochlorine waste streams	Very dark (opaque) viscous liquid	17	17
2E	Wastewater from storage tank	Tan liquid (very small amount of dark brown liquid on top)	106	10,24,106
3E	Waste naphtha	Light brown clear liquid	16,28, or 29	4,16,31
4E	Carbon tetrachloride	Dark brown clear liquid	17	3,16,17
5E	MIBK with waste solvents	Light pink clear liquid	19	4,19,31

(continued)

[a] Sources of hazardous waste samples are being kept anonymous at the request of the project technical advisor

[b] See discussion

[c] Not well defined by waste description

[d] Also found to contain organics

[e] Water not predicted by waste description

TABLE 21. (concluded)

Sample[a]	Sample identification	Sample description	RGN(s) Predicted	RGN(s) Found
6E[b]	Waste solvents with amines	Light brown clear liquid	7	10,24,106[b]
7E	Thiocarbohydrazide with H_2S	Red-orange clear liquid	33,105,106	10,33,105,106
8E	Spent caustic	Clear colorless viscous liquid with small amount of black particulate	10,106	10,106
9E	Ethylene dichloride still bottoms	Very dark blue liquid	17	17
10E	Waste solvents, waste ink	Dark gray viscous liquid	b	13,19
11E	Waste varnish	Light brown clear viscous liquid	16	16
12E	Water base insecticide	Milky white opaque viscous suspension	106[c]	106[d]
13EU	Oil base insecticide	Yellow clear liquid (upper layer of two layers)	d	16
13EL	Oil base insecticide	Cloudy light yellow liquid (lower layer of two layers)	d,e	106
14E	Tribromocumene	Opaque red-brown viscous suspension	16,17	16,17
15E	PCB oil, high level	Light yellow-green clear liquid	16,17	16,17
16E	PCB oil, low level	Light orange clear liquid	16,17	16
17E	Silicone oil (with PCB's)	Very light yellow-green clear liquid	16,17	16

[a] Sources of hazardous waste samples are being kept anonymous at the request of the project technical advisor

[b] See discussion

[c] Not well defined by waste description

[d] Also found to contain organics

[e] Water not predicted by waste description

TABLE 22. DETAILED RESULTS OF FIELD TESTING OF HAZARDOUS WASTE SAMPLES

Sample	Results of testing						RGN(s) expected	RGN(s) found	Additional comments
	PS 1[a]	PS 2	PS 3	PS 4	PS 5	PS 6[c]			
1D	N/N	Soluble organic; insoluble H_2O, acids; S, CN, H_2O negative	Burns, organic	X, 0	4[d]	--[b]	17	4,17	
2D	N/N	Soluble organic; insoluble H_2O, acids; S, CN, H_2O negative	Crackles, white smoke	X only	16	--	17,(16,28,29)	16,17	Explosive?
3DU	N/N	Soluble organic; insoluble H_2O, acids; S, CN, H_2O negative	Burns, organic	None	16	--	4,(16,28,29)	16	Ethanol soluble; not found in lower (aqueous) layer
3DL	N/N	Soluble H_2O, acids; insoluble organic; S, CN negative; H_2O positive	Evaporates, residue, inorganic	--	--	M negative F negative	4,(106)	106	Mirror, organic metal
4DU	N/N	Soluble organic, H_2O, acids; S, CN, H_2O negative	Burns, organic	0 only	19 (+16)	M negative F negative	16,19	(106), 16, 19	Hydrocarbon probably present
4DL	N/N	Soluble H_2O, acids; insoluble organic; S, CN negative, H_2O positive	Evaporates, brown residue, inorganic	--	--	M negative F negative	106	106	
5D	N/N	Soluble Ac, MeOH; insoluble hexane, toluene, H_2O, acids; (soluble H_2SO_4) S, CN, H_2O negative	Burns, organic, smoke, residue	N, 0	19, 4; no N RGN's found	--	16,19	4,16,19	Appears to be paint and residue, N present but all N RGN's negative
6D	N/N	Soluble organic; insoluble H_2O, acids; S, CN, H_2O negative	Burns, organic, residue	0 only	19	--	19	19	Appears to be paint residue
7D	N/N	Soluble organic; insoluble H_2O; darkens acid; S, CN, H_2O negative	Burns, organic, residue	N, 0	19 no N RGN's found	--	19	16,19	Hydrocarbon probably present

[a] Expressed as acidity/redox (i.e., B/R-base, reducing; N/N neutral, nonoxidizing and nonreducing; A acid)

[b] -- not done

[c] M = metal
F = fluoride

[d] Numbers refer to predicted RGN

(continued)

TABLE 22. (continued)

Sample	Results of testing						RGN(s) expected	RGN(s) found	Additional comments
	PS 1[a]	PS 2	PS 3	PS 4	PS 5	PS 6[c]			
8DU	N/N	Soluble toluene, hexane; insoluble Ac, H_2O, acids; S, CN, H_2O negative	Crackles, burns, organic	--[b]	--	--	??	102	
8DL	A(4)/N	Soluble H_2O, acids; insoluble organic; S, CN negative; H_2O positive	Evaporates, residue, inorganic	--	--	M positive F negative	??	24,106	
1E	N/N	Soluble Ac, toluene; insoluble H_2O, acids; S, CN, H_2O negative	Evaporates, then burns	X, N (0)	No N RGN's found		17	17	Probably also contains hydrocarbons
2E	B(11)/N	Soluble H_2O, acids, Ac, MeOH; insoluble toluene, hexane; S, CN negative; H_2O positive	Boils, gray residue, inorganic	--	--	M positive F negative	106	10,24, 106	
3E	N/N	Soluble toluene hexane; insoluble H_2O, acid, Ac, MeOH; S, CN, H_2O negative	Burns	S(0)	4,31[d]	--	16,28 or 29	4 and/or 16,31	S present but no S RGN's identified
4E	A(4)/N	Soluble organic; insoluble acids, H_2O; S, CN, H_2O negative	Burns, yellow, organic	X, 0	3,16	--	17	3,16,17	Violent explosion during sodium fusion
5E	N/N	Soluble organic, acids; insoluble H_2O; S, CN, H_2O negative	Does not burn	0 only	4,19,31	--	19	4,19,31	
6E	B(12)/N	Soluble H_2O, acids; Ac, MeOH; insoluble toluene, hexane; S, CN negative; H_2O positive	Boils, gray residue, inorganic	--	--	M positive F negative	7	10,24,106	

(continued)

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[a] Expressed as acidity/redox (i.e., B/R-base, reducing; N/N neutral, nonoxidizing and nonreducing; A acid)

[b] -- not done

[c] M = metal
F = fluoride

[d] Numbers refer to predicted RGN

TABLE 22. (continued)

Sample	Results of testing PS 1[a]	PS 2[b]	PS 3	PS 4	PS 5	PS 6[c]	RGN(s) expected	RGN(s) found	Additional comments
7E	B(>10)/R	Solubilities not done; S, H_2O positive; CN negative	--	--	--	--	33,105,106	10,33, 105,106	
8E	B(12)/N	Insoluble organic; reacts violently with acids, H_2O present; S, CN negative	Boils, gray, inorganic	- -	--	M negative F negative	10,106	10,106	Unusual material, reactive
9E	N/N	Soluble organic; insoluble H_2O, acids; S, CN, H_2O negative	Burns, smoke, residue, organic	X only	17 only	--	17	17	
10E	N/N	Soluble organic, H_2O, acids	Burns	N(0) positive	13,5	--	??	6,13,19	Looks like black paint, difficult to observe, dark sample
11E	N/N	Soluble organic; insoluble acids; S, CN, H_2O negative	Burns, organic	None	16 only	--	16	16	
12E	B(10)/N	Soluble H_2O, acid, Ac, MeOH; insoluble toluene, hexane; S, CN negative; H_2O positive	Burns, slightly	--	19 only	M negative F negative	106	19,106	Water soluble organic
13EU	N/N	Soluble H_2O, acid, Ac, MeOH; insoluble toluene, hexane; S, CN negative; H_2O positive	Burns, organic	None	16 only	--	16,106	16,106	
13EL	N/N	Soluble H_2O, acid, Ac, MeOH; insoluble toluene, hexane; S, CN negative; H_2O positive	Boils, does not burn	--	--	M negative F negative	16,106	106	
14E	N/N	Soluble organic; insoluble H_2O, acids; S, CN, H_2O negative	Burns	X only	16,17	--	16, 17	16,17	

(continued)

[a] Expressed as acidity/redox (i.e., B/R-base, reducing; N/N neutral, nonoxidizing and nonreducing; A acid)

[b] -- not done

[c] M = metals
F = fluoride

[d] Numbers refer to predicted RGN

TABLE 22. (concluded)

Sample	Results of testing						RGN(s) expected	RGN(s) found	Additional comments
	PS 1[a]	PS 2[b]	PS 3	PS 4	PS 5	PS 6			
15E	N/N	Soluble organic; insoluble H_2O, acids; S, CN, H_2O negative	Burns	X only	16, 17	--	16, 17	16, 17	
16E	N/N	Soluble toluene, hexane; insoluble H_2O, acids, Ac, MeOH; S, CN, H_2O negative	Burns	None	16 only	--	16, 17	16	
17E	N/N	Soluble toluene, hexane, Ac; insoluble H_2O, acid, MeOH; S, CN, H_2O negative	Burns	None	16 only	--	16, 17	16	

---

[a] Expressed as acidity/redox (i.e., B/R-base, reducing; N/N neutral, nonoxidizing and nonreducing; A acid)

[b] -- not done

[c] M = metal
F = fluoride

[d] Numbers refer to predicted RGN

sulfur and the specific functionality tests. The same conclusion can be reached from the results of sample 10E. A positive nitrogen response was obtained but no nitrogen compounds were detected in this waste sample.

A carbon tetrachloride waste sample (4E) was found to contain aromatics and organic acids as well as being in RGN 17 (chlorinated organics).

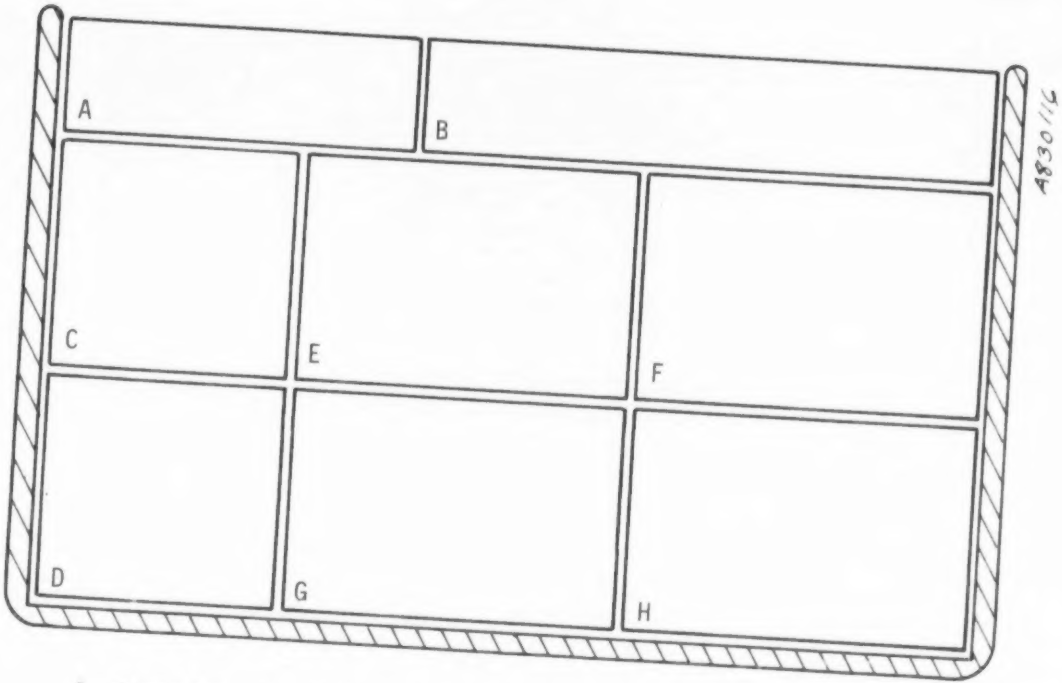
Two minor shortcomings of the test schemes were pointed out by the field evaluation. The test schemes are organized such that aqueous liquids are not tested for organics. The sodium fusion procedure, as well as a number of specific organic functionality tests are not amenable to aqueous solutions. For compatibility purposes, the classification of aqueous organics as aqueous mixtures would probably suffice. For purposes of predicting optimum destruction technology, the inability to detect organics in water may be a serious limitation.

During the field test, approximately 40 man-hours were spent in actual sample analysis. Since 29 phases were analyzed, an average analysis time of approximately 1.3 hr/sample was realized. This average analysis time is expected to be quite acceptable in real-world field applications.

3.8 ASSEMBLY OF FIELD TEST KIT

The field test kit contains all equipment, reagents, and safety devices necessary for carrying out all tests as specified in the test manual.

The field test kit was designed to meet the following requirements: small volume and few containers for ease of transportation, easy access to all test materials and equipment, and prevention of leaking of reagents and breakage of equipment during transport. Smaller items such as spatulas, spoons, burner accessories, spotplates, beakers, etc. are put into larger containers which were labeled to facilitate identification of contents. The specific equipment and reagents used in the test kit and their distribution in the coolers are shown in Figures 9, 10, and 11. For transportation purposes all test equipment was placed into three 13-in. by 22-in. by 15-in. Coleman coolers. Other containers of similar dimensions and durability can be used also. For safe transportation and to organize the reagents according to procedure sets, all reagents required for the tests within a procedure set were placed together in a specially designed redwood box. Wood was used because of its ruggedness, its resistance to most solvents and acids, and because a support with holes matching the different diameters of the reagent bottles could be manufactured with simple tools at relatively low cost. The kit includes four reagent cases, one each for PS 1, PS 2, PS 6; PS 4, PS 5A, PS 5B; and PS 5C, PS 5D. A similar box, with a foam insert was constructed to hold the mixing device (see Figure 11). The reagent boxes were packed in cooler 1 together with some small equipment and supplies; larger equipment including safety shield, test tubes, mixing device, beakers, and funnels were packed in cooler 2; and safety equipment (coveralls, rubber and latex gloves, respirator, goggles), paper towels, and propane tanks were put in cooler 3. Water- and alcohol-based reagents were placed in 3-oz. polyethylene reagent dropping bottles; and noncorrosive solids and test papers were placed in



A Spatulas, ceramic spoons, wire loops, glass rods, test tube holders, pipet bulbs

B Lighter, flame tips, stand for propane torch, forceps, wire screens, scissors, pliers, screwdriver

C Test tube rack and 4-in. test tubes

D Test tube rack and 3-in. test tubes

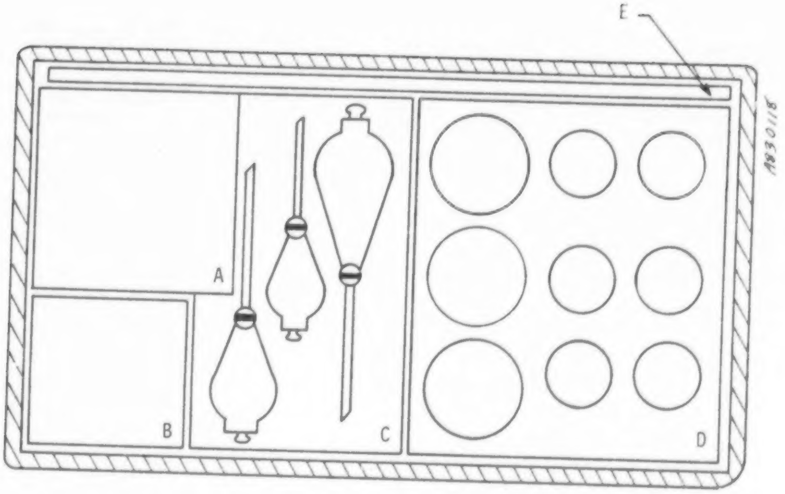
E Reagents for PS 1, PS 2, PS 6

F Reagents for PS 4, PS 5A

G Reagents for PS 5B

H Reagents for PS 5C, PS 5D

Figure 9. Cooler 1 -- equipment organization.



A Pasteur pipets

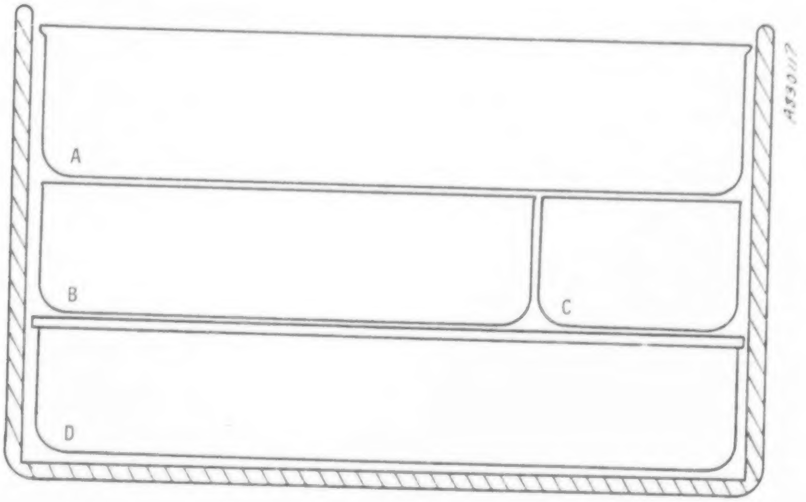
B Spotplates

C Separatory funnels

D Beakers

E Safety shield

Figure 10a. Cooler 2 -- equipment organization top tray.



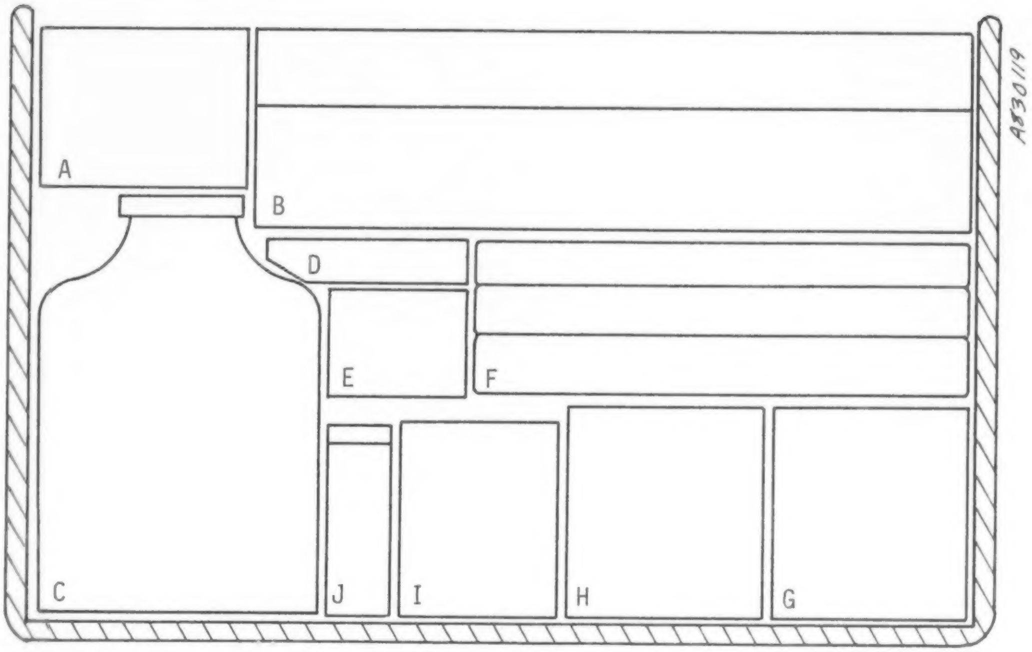
A Pipets, spotplates, funnels, beakers

B Filter paper, watchglasses, parafilm, extra sample vials

C Pencils, pens, markers, labels, tape

D Clamp, stand, ring support, kemwipes, paper towels, pipets

Figure 10b. Cooler 2 -- equipment organization.



A Latex gloves

B Mixing device

C Wash solvents (acetone, methanol)

D Acid gloves

E Respirators, goggles

F Safety suits

G Rack with sample vials

H Test tubes, 4 in.

I Test tubes, 3 in.

J Sodium hydroxide and barium nitrate solutions

K Propane tanks (2) } Behind cutaway

L Glycerol }

Figure 11. Cooler 3 -- equipment organization.

30-mL polyethylene screw cap bottles. Corrosive liquids, and reagents dissolved in hexane, toluene, acetone, and chloroform as well as the neat solvents are placed in 30-mL Teflon dropping bottles. For shipment all dropping caps were replaced with regular screw caps.

The test kit arrived in perfect condition without any reagent spills at the field test site after shipment by air cargo.

3.9 SAFETY

Safe handling of reagents and hazardous waste samples is of paramount importance to field testing operations. It is assumed that analysts will be trained in the safe handling of hazardous waste materials. Some key safety principles are discussed in the following paragraphs. The first section deals with generalities regarding safe operation of the field test kit. The second section describes safe handling of hazardous waste samples and the third describes safe handling of equipment and reagents contained within the test kit. Finally, a short section on safe transportation of the test kit is included.

General Safety Precautions

The safe performance of the field test procedures depends primarily upon the use of very small amounts of materials, such that undesirable effects of reactions resulting from the test procedures are kept to a minimum. For this reason, the tests should be conducted with the smallest quantities of test materials feasible, and only small samples of the waste materials (i.e., 50g or less) should be kept at the testing site. Since several of the test procedures include the use of a flame and reactive reagents, it is essential that tests be performed in an area well removed from bulk storage areas.

Personal protective clothing and devices are provided with the test kit. These include: safety glasses and goggles, latex exam gloves and heavy viton gloves, Tyvac coveralls, and half-face respirators. Gloves and eye protection are mandatory, and the use of other personal protective devices is very strongly encouraged any time samples or reagents are being handled or tests are being conducted. In addition, a portable safety shield is provided as an added margin or safety for conducting tests and should be employed, particularly for the sodium fusion procedure and other procedures that involve heating of waste materials.

Handling of Hazardous Waste Samples

Hazardous waste samples may contain virtually any stable chemical element or compound known and most are complex mixtures. For the present context it is assumed that no prior knowledge of the waste material is available. Waste materials may present any or several of the following types of hazards:

- Toxic
- Explosive
- Extremely corrosive
- Violently reactive when mixed with certain other substances (e.g., water-reactive materials)
- Materials which emit toxic or explosive fumes
- Biologically hazardous materials (e.g., biological warfare agents)
- Radiologically hazardous materials
- "Exotic" chemical toxins (e.g., chemical warfare agents)

The present testing sequence does not address the problems of biologically hazardous materials, radioactive materials, or exotic chemical toxins. Normally, prior knowledge of the presence of these materials will be available and further special precautions will have to be taken.

Extreme caution is mandatory when sampling drums for field testing. Although the methodology described herein does not describe drum sampling, the safe collection of samples for testing is nonetheless worthy of mention here. Drums are opened in an open area removed as much as possible from bulk storage areas. Personnel collecting the samples are outfitted with full protective clothing, including heavy gloves. Samples are collected using dip-tubes or other suitable means and transferred to medium-size containers. For testing, small aliquots of the samples are transferred to small containers and care is taken to wipe the exterior of the sample container free of any contamination from the sample.

Safe Conduct of Field Tests

Tests are conducted in an open, well-ventilated area removed from bulk storage areas for drums. Protective clothing and devices are used, as discussed in the previous sections. Small amounts of test materials and reagents are employed to minimize the effects of potentially catastrophic reactions.

Reagents employed in the test kit are hazardous in themselves. The specific hazards presented by the reagents in the kit are as listed in Table 23.

In addition to the hazards specified above, most of the dissolved reagents are toxic and most of the reagents should be treated as potential carcinogens.

Wastes from the test procedures should be treated as hazardous (they are probably more hazardous than the waste materials themselves) and allowance

TABLE 23. HAZARDS OF TEST KIT REAGENTS

Reagent(s)	Procedure set(s) where employed	Hazard presented
Acetone, methanol, toluene, hexane	PS 2, 4	Flammable liquid
Hydrochloric, sulfuric acid	PS 2	Corrosive liquid
Nitric acid	PS 2, 4, 6	Corrosive liquid oxidizer
Sodium hydroxide	PS 2	Caustic
Propane	PS 3	Compressed gas, flammable
Diethyl ether	PS 4, 5	Extremely flammable, volatile liquid
Sodium metal	PS 4	Water-reactive flammable solid strong reducing agent
Phenylhydrazine	PS 5	Strong reducing agent, corrosive
Iodine/carbon disulfide	PS 5	Extremely flammable, oxidizing, volatile liquid
Stannous chloride	PS 5	Strong reducing agent
Ethanol	PS 5	
Potassium hydroxide/methanol	PS 5	Caustic, flammable liquid
Hydroxylamine hydrochloride	PS 5	Strong reducing agent
Potassium permanganate/acetone	PS 5	Strong oxidizer, flammable
Aluminum chloride	PS 5	Water-reactive solid
Phosphomolybdic acid	PS 6	Strong oxidizing agent, corrosive liquid
Zinc sulfide, ammonium sulfide	PS 6	Emits toxic gas (H_2S)

should be made for proper disposal. These waste materials can be placed in polyethylene containers placed in a drum and surrounded by vermiculite or other absorptive material.

Safe Transportation of the Test Kit

The test kit is designed to withstand the rigors of shipping. Hazardous materials are contained in unbreakable containers. Materials which adversely affect polyethylene are stored in Teflon bottles. The containers are fitted with solid caps for shipping; these are replaced with dropper caps at the testing site. Glass vessels are protected from breakage.

The specific hazards presented by many of the reagents are presented in the preceeding section. Many of these materials may not be shipped in the same container. For example, acids may not be shipped in the same container with flammable liquids. For this reason, acids and other corrosives are shipped in one cooler and all flammable liquids shipped in a second cooler. Metallic sodium and nitric acid must both be shipped separately; carbon disulfide cannot be shipped by air.

The above regulations apply to commercial (air cargo) shipping. It is recommended that, for reasons of convenience in transportation, the field test kit be transported by private vehicle when possible.

REFERENCES

*1. Hatayama, H. K., J. J. Chen, E. R. de Vera, R. D. Stephens, and D. L. Strom, "A Method of Determining the Compatibility of Hazardous Wastes," EPA/600/2-80-076, U.S. Environmental Protection Agency, Cincinnati, Ohio, 1980, NTIS PB No. 80-221005.

2. Cheronis, N. D., and J. B. Entrikin, "Semimicro Qualitative Organic Analysis," Interscience Publishers, Inc., New York, New York, 1960.

3. Feigl, F., "Spot Tests in Inorganic Analysis," Elsevier Publishing Company, New York, New York, 1972.

4. Feigl, F., "Spot Tests in Organic Analysis," Elsevier Publishing Company, New York, New York, 1966.

5. Rand, H. C., A. E. Greenberg, and M. J. Taras, "Standard Methods for the Examination of Water and Wastewater," 14th ed., American Public Health Association, Washington, D.C., 1978.

6. Simmons, B. P., I. Tan, T. H. Li, R. D. Stephens, and D. L. Strom, "A Method for Determining the Reactivity of Hazardous Wastes," U.S. Environmental Protection Agency, Cincinnati, Ohio, 1982 (Preliminary).

7. Flynn, J. P. and H. E. Rossow, "Classification of Chemical Reactivity Hazards," Dow Chemical Co., Midland, Michigan, December 1970 for National Academy of Science and U.S. Coast Guard, USCGD 29-74, NTIS AD-733 049.

*This EPA report is no longer available from EPA or NTIS. An updated version of this report entitled "A Proposed Guide for Estimating the Incompatibility of Selected Hazardous Wastes Based on Binary Chemical Reactions" is scheduled to be published in 1984 by the American Society for Testing and Materials (ASTM) D34 Committee on Waste Disposal.

APPENDIX

FIELD TEST KIT INSTRUCTION MANUAL

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A.1 INTRODUCTION AND SAFETY

This manual describes the use of the Hazardous Waste Classification Field Test Kit developed for the U.S. Environmental Protection Agency (EPA) by Acurex Corporation. The development of the test kit and verification of the test procedures is described in EPA Report ___________, "Hazardous Waste Compatibility Testing for Applicability to Remedial Action at Uncontrolled Hazardous Waste Sites."

A.1.1 Scope

The tests described herein are intended to provide qualitative information regarding the bulk chemical composition of hazardous waste materials. The test procedures are used to classify waste materials into reactivity groups (RGN's) defined in EPA Report 600/2-80-076, "A Method for Determining the Compatibility of Hazardous Wastes." The information thus obtained is intended to be used to determine compatibility characteristics of hazardous wastes, in conjunction with the compatibility chart in EPA-600/2-80-076. But it may be used for other purposes such as hauler manifest verification or prediction of best disposal technology. These test procedures do not address the potential of radioactive or biological hazards. It should also be stressed that these procedures provide only qualitative information without regards to concentration. Therefore, a specific hazard (i.e., sulfide) will be identified even at a level such that it poses a minimal hazard. To this extent, the test results may lead to misleadingly conservative conclusions. Conversely, these procedures will probably not identify materials at low concentrations (Ca <5 percent) that may cause long-term hazards. The lower limits of response for the various tests have not been determined.

Preliminary procedures are provided to give information regarding general reactivity characteristics of waste materials such as acidity/basicity, oxidative/reductive potential, reactivity/solubility characteristics, and combustibility. Additional procedures are provided as specific class tests for complete categorization of waste materials according to chemical functionality. Any or all of these procedures can be employed, depending upon the discretion of a qualified supervisor and the completeness of information required.

The kit and manual are designed for use by technicians with minimal knowledge of the meaning of the results. The analyst is expected to have had some limited laboratory experience and at least 1 week of hands-on training. The test procedures have been designed to limit judgment decisions to a minimum.

A simple device to detect heat and gas evolution upon mixing of two waste materials is included in the test kit. It is strongly recommended that this device be used to test the compatibility of the wastes even if the compatibility chart indicates the wastes to be compatible.

On the other hand, completion of the testing sequence prior to mixing is strongly recommended for the following reasons:

- To identify hazards that may be dangerous upon mixing even in small quantities
- To identify long-term adverse effects of mixing not observed in the mixing device but predicted by the compatibility chart
- To utilize the classification information obtained in testing for purposes other than compatibility prediction (e.g., identification of specific wastes or waste stream categories)

The test procedures described herein are organized into six series of tests, each included to provide specific information about the waste material. In several cases, results obtained at a specific stage in the testing sequence can sufficiently define the chemical characteristics of the waste material such that the need for further testing is eliminated. These cases are pointed out in the discussion of the procedure sequences. Each of the six procedure sets is summarized with a flow diagram, which provides an overview of procedures involved in that particular procedure set.

Section A.2 of this manual includes a presentation and discussion of the procedure sets, and generalized instructions on the use of the test sequence. Section A.3 includes a test data sheet and instructions for its use. Section A.4 contains specific instructions for conducting each of the test procedures, including instructions for preparation of reagents. A description of testing to determine the effects of mixing wastes is presented in Section A.5. Lists of equipment and supplies are included as attachments.

Specific test procedures are provided for classification of waste materials into the RGN's listed in Table A-1. In many cases specific classification may not be required (i.e., distinction between aliphatic and aromatic hydrocarbons). In those cases where, in the opinion of the authors, tests are not absolutely required for purposes of defining compatibility characteristics, the procedures are labeled optional (and are indicated by broken lines on the flow diagrams). Likewise, when, in the opinion of the authors, further testing may present a hazard (i.e., sulfides, cyanides), a "stop" is indicated in the flow diagrams.

A.1.2 Safety

Safe handling of reagents and hazardous waste samples is of paramount importance to field testing operations. It is assumed that analysts will be trained in the safe handling of hazardous waste materials. Some key safety principles are discussed in the following paragraphs. The first subsection deals with generalities regarding safe operation of the field test kit. The second subsection describes safe handling of hazardous waste samples and the third describes safe handling of equipment and reagents contained within the test kit.

Table A-1. REACTIVITY GROUP DEFINITIONS

Reactivity group no.	Reactivity group name
1	Acids, mineral, nonoxidizing
2	Acids, mineral, oxidizing
3	Acids, organic
4	Alcohols and glycols
5	Aldehydes
6	Amides
7	Amines, aliphatic and aromatic
8	Azo compounds, diazo compounds, and hydrazines
9	Carbamates
10	Caustics
11	Cyanides
12	Dithiocarbomates
13	Esters
14	Ethers
15	Fluorides, inorganic
16	Hydrocarbons, aromatic
17	Halogenated organics
18	Isocyanates
19	Ketones
20	Mercaptans and other organic sulfides
21	Metals, alkali and alkaline earth, elemental and alloys
22	Metals, other elemental and alloys in the form of powders, vapors, or sponges
23	Metals, other elemental and alloys as sheets, rods, moldings, drops, etc.
24	Metals and metal compounds, toxic
25	Nitrides
26	Nitriles
27	Nitro compounds
28	Hydrocarbons, aliphatic, unsaturated
29	Hydrocarbons, aliphatic, saturated
30	Peroxides and hydroperoxides, organic
31	Phenols and cresols
32	Organophosphates, phosphothioates, phosphodithioates
33	Sulfides, inorganic
34	Epoxides
101	Combustible and flammable materials, miscellaneous
102	Explosives
103	Polymerizable compounds
104	Oxidizing agents, strong
105	Reducing agents, strong
106	Water and mixtures containing water
107	Water reactive substances

A.1.2.1 General Safety Precautions--

The safe performance of the field test procedures depends primarily upon the use of very small amounts of materials, such that undesirable effects of reactions resulting from the test procedures are kept to a minimum. For this reason, the tests should be conducted with the smallest quantities of test materials feasible, and only small samples of the waste materials (i.e., 50g or less) should be kept at the testing site. Since several of the test procedures include the use of a flame and reactive reagents, it is essential that tests be performed in an area well removed from bulk storage areas.

Personal protective clothing and devices are to be provided with the test kit. These include: safety glasses and goggles, latex exam gloves and heavy viton gloves, Tyvac coveralls, and half-face respirators. Gloves and eye protection are mandatory and the use of other personal protective devices is very strongly encouraged any time samples or reagents are being handled or tests are being conducted. In addition, a portable safety shield is provided as an added margin of safety for conducting tests and should be employed particularly for the sodium fusion procedure and other procedures where heating of waste materials is involved.

Emergency procedures and facilities at the site must be identified before testing is initiated. As a minimum, the phone numbers of the nearest fire and medical facilities should be posted, along with directions to the nearest emergency facilities.

A.1.2.2 Handling of Hazardous Waste Samples--

Hazardous waste samples may contain virtually any stable chemical element or compound known and most are complex mixtures. For the present context it is assumed that no prior knowledge of the waste material is available. Waste materials may present any or several of the following types of hazards:

- Toxic
- Explosive
- Extremely corrosive
- Violently reactive when mixed with certain other substances (i.e., water-reactive materials)
- Materials which emit toxic or explosive fumes
- Biologically hazardous materials (i.e., biological warfare agents)
- Radiologically hazardous materials
- "Exotic" chemical toxins (i.e., chemical warfare agents)

The present testing sequence does not address the problems of biologically hazardous materials, radioactive materials, or exotic chemical toxins. Normally, prior knowledge of the presence of these materials will be available and further special precautions will have to be taken.

Extreme caution is mandatory when sampling drums for field testing. Although the methodology described herein does not describe drum sampling, some aspects of the safe collection of samples for testing is nonetheless worthy of mention. Drums should be opened in an open area removed as much as possible from bulk storage areas. Personnel collecting the samples should be outfitted with full protective clothing, including heavy gloves. Samples should be collected using dip tubes or other suitable means and transferred to medium-size containers. For testing, small aliquots of the samples should be transferred to small containers (approximately 5 mL) and care should be taken to wipe the exterior of the sample container free of any contamination from the sample.

A.1.2.3 Safe Conduct of Field Tests--

Tests should be conducted in an open, well-ventilated area removed from bulk storage areas for drums. Protective clothing and devices should be employed, as discussed in the previous sections. Small amounts of test materials and reagents are employed to minimize the effects of potentially catastrophic reactions.

Reagents employed in the test kit are hazardous in themselves. The specific hazards presented by the reagents in the kit are presented in Table A-2.

In addition to the hazards specified in Table A-2, most of the dissolved reagents are toxic and most of the reagents should be treated as potential carcinogens.

Wastes from the test procedures should be treated as hazardous (they are probably more hazardous than the waste materials themselves) and allowance should be made for proper disposal. These waste materials can be placed in polyethylene containers placed in a drum and surrounded by vermiculate or other absorptive material. These waste materials will fall into the regulatory category of laboratory wastes.

A.2 PROCEDURE SEQUENCES

A.2.1 Sequence of Procedure Sets

The procedures employed for chemical categorization of hazardous wastes are organized into six procedure sets (PS), briefly summarized in Table A-3.

The first step in the classification of an unknown hazardous waste material is the physical (visual) examination of the waste material. The physical state of the material (solid, liquid, etc.) may provide considerable insight as to its identity (i.e., metal castings). Depending upon the physical state of the material, the procedure sets are performed in the sequence indicated in Figure A-1.

TABLE A-2. REAGENTS AND HAZARDS

Reagent(s)	Procedure set(s) where employed	Hazard presented
Acetone, methanol, toluene, hexane	PS 2, 4	Flammable liquid
Hydrochloric, sulfuric acid	PS 2	Corrosive liquid
Nitric acid	PS 2, 4, 6	Corrosive liquid; oxidizer
Sodium hydroxide	PS 2	Caustic
Propane	PS 3	Compressed gas; flammable
Diethyl ether	PS 4, 5	Extremely flammable, volatile liquid
Sodium metal	PS 4	Water-reactive, flammable solid; strong reducing agent
Phenylhydrazine	PS 5	Strong reducing agent
Iodine/carbon disulfide	PS 5	Extremely flammable, oxidizing, volatile liquid
Stannous chloride	PS 5	Strong reducing agent
Ethanol	PS 5	
Potassium hydroxide/ methanol	PS 5	Caustic, flammable liquid
Hydroxylamine hydrochloride	PS 5	Strong reducing agent
Potassium permanganate/ acetone	PS 5	Strong oxidizer, flammable
Aluminum chloride	PS 5	Water-reactive solid
Phosphomolybdic acid	PS 6	Strong oxidizing agent, corrosive liquid
Zinc sulfide, ammonium sulfide	PS 6	Emits toxic gas (H_2S)

Table A-3. PROCEDURE SETS

PS	Title	Information obtained
1	pH and Redox Tests	Acidity, basicity, oxidizing and reducing potential
2	Solution-Reactivity and Special Functionality Tests	Identification of sulfides and cyanides, reactivity and solubility in acids and solvents, reactivity with water, presence of water
3	Flame Test	Combustibility, classification as organic or inorganic, identification of explosives
4	Sodium Fusion and Ferrox Tests	Identification of oxygen, nitrogen, phosphorus, sulfur, and halogen in organic waste materials
5	Organic Functionality Tests	Presence of specific organic functional groups
6	Inorganic Functionality Tests	Presence of elemental metals, heavy metal compounds, and inorganic fluorides

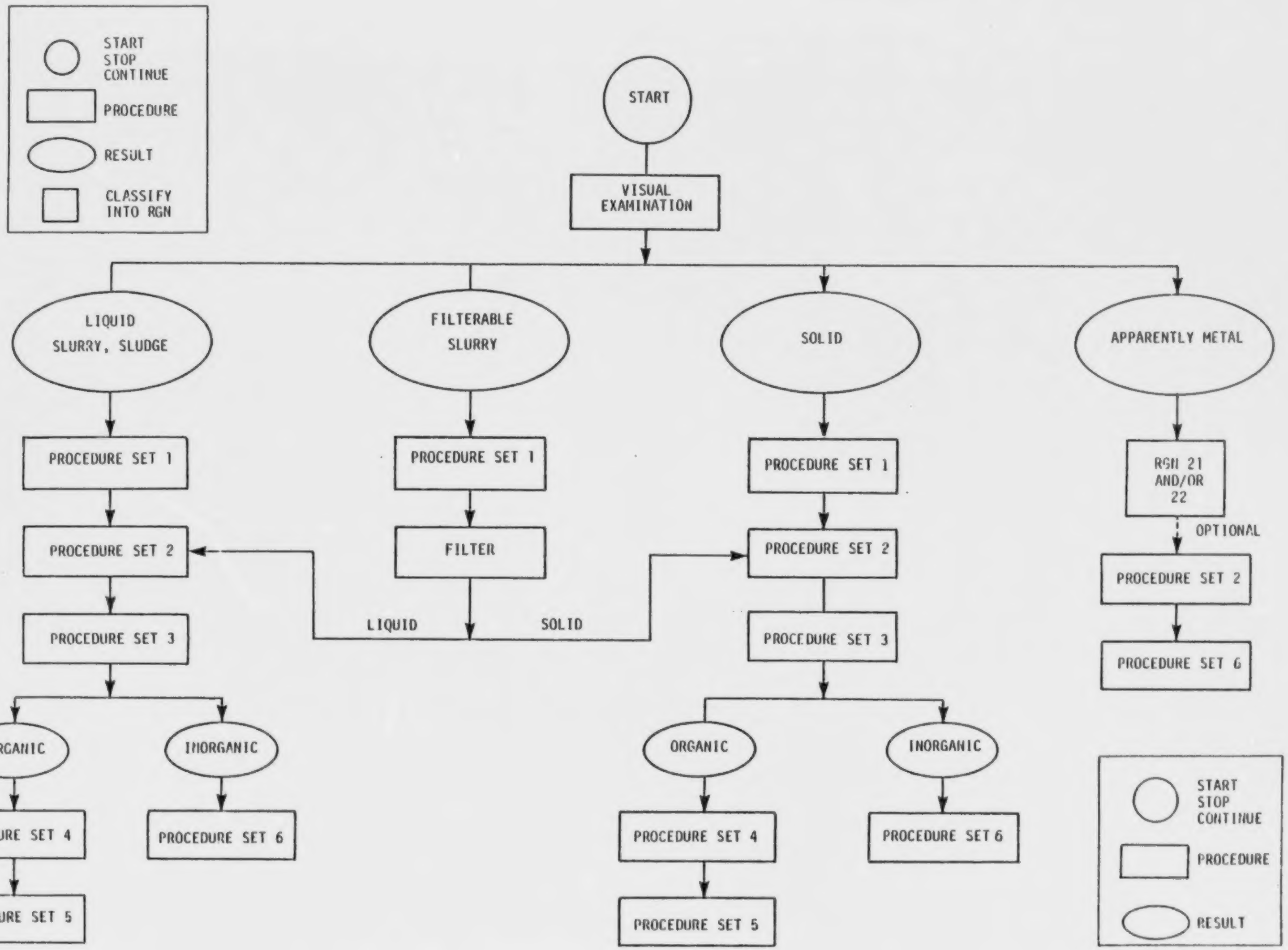

START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY
INTO RGN
START
VISUAL
EXAMINATION
LIQUID
SLURRY, SLUDGE
FILTERABLE
SLURRY
SOLID
APPARENTLY METAL
PROCEDURE SET 1
PROCEDURE SET 1
PROCEDURE SET 1
RGN 21
AND/OR
22
OPTIONAL
PROCEDURE SET 2
FILTER
PROCEDURE SET 2
PROCEDURE SET 2
PROCEDURE SET 3
LIQUID
SOLID
PROCEDURE SET 3
PROCEDURE SET 6
ORGANIC
INORGANIC
ORGANIC
INORGANIC
PROCEDURE SET 4
PROCEDURE SET 6
PROCEDURE SET 4
PROCEDURE SET 6
PROCEDURE SET 5
PROCEDURE SET 5
START
STOP
CONTINUE
PROCEDURE
RESULT

Figure A-1. Sequence of procedure sets.

Procedure set 1 (PS 1) provides considerable information in a very short period of time and thus is performed first. PS 1 may be performed concurrently with PS 2, for convenience if desired. Next, the combustibility of the material is observed by performing the flame test (PS 3) which should be conducted, even if not indicated from PS 2, to detect potentially explosive materials. The results of PS 2 and PS 3 are used to determine whether the material is primarily organic in nature (combustible) or primarily inorganic. Generally, flammable materials or materials that are solvent-soluble are classified as organic and materials that fuse or evaporate are classified as inorganic. Appropriate functionality tests are applied to fully characterize the material, depending upon its classification as organic or inorganic. For the purposes of the tests employed herein, slurries and sludges are treated in the same manner as liquids.

It is recommended, where possible, that slurries be filtered and the solid and liquid portions be analyzed separately. Otherwise, unless specified, separate portions of untreated waste materials are used for each of the tests. When two or more liquid phases exist, it is advisable to test each phase as a different sample.

A.2.2 Procedure Set Descriptions

Flow diagrams for procedure sets 1 through 6 are shown in Figures A-2 through A-7, and are explained in detail in Sections A.2.2.1 through A.2.2.6. Specific instructions for conducting the test procedures are given in Section A.4.

A.2.2.1 PS 1 -- pH and Redox Tests--

Oxidation, reduction, and pH tests are performed on a waste material by means of test papers. These tests identify some of the most reactive materials and therefore are performed early in the testing sequence.

Acids (RGN's 1, 2, 3) or bases (caustics -- RGN 10) are identified by means of the pH test. Mineral acids are identified by a pH of 1.5 or less. Organic acids are so designated by pH of 2 to 5. Bases (caustics) have a pH of 10.5 to 14. Oxidizing agents (RGN 104) and reducing agents (RGN 105) are determined by similar paper tests.

Classification of a waste material into RGN's 1, 2, or 104 adequately defines the waste material such that further testing is unnecessary. Also, if the material is caustic (RGN 10) and a reducing agent (RGN 105) further testing is unnecessary, as indicated in Figure A-2. Performance of the flame test to identify explosives is recommended, however, as indicated in Figure A-2. The procedures used for these tests are described in Section A.4.1.

A.2.2.2 PS 2 -- Solution-Reactivity and Special Functionality Tests--

The waste material is treated with water, a base, and with various acids and solvents to determine its reactivity and solubility characteristics. The flow diagram for PS 2 is shown in Figure A-2. The first step is treatment with water. Materials which are water reactive (RGN 107) (as indicated by

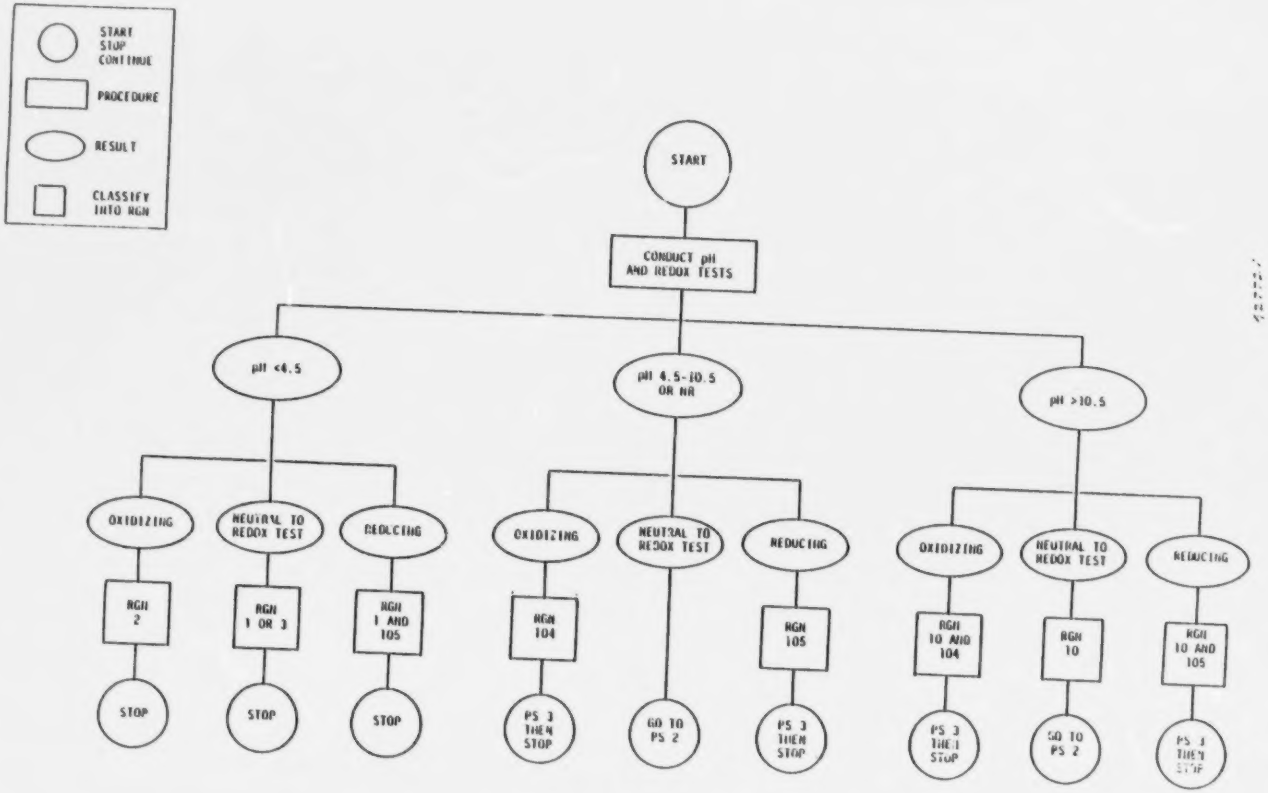

START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY
INTO RGN
START
CONDUCT pH
AND REDOX TESTS
pH <4.5
pH 4.5-10.5
OR NR
pH >10.5
OXIDIZING
NEUTRAL TO
REDOX TEST
REDUCING
RGN
2
RGN
1 OR 3
RGN
1 AND
105
STOP
STOP
STOP
OXIDIZING
NEUTRAL TO
REDOX TEST
REDUCING
RGN
104
RGN
105
PS 3
THEN
STOP
GO TO
PS 2
PS 3
THEN
STOP
OXIDIZING
NEUTRAL TO
REDOX TEST
REDUCING
RGN
10 AND
104
RGN
10
RGN
10 AND
105
PS 3
THEN
STOP
GO TO
PS 2
PS 3
THEN
STOP

Figure A-2. Procedure set 1 -- pH and redox tests.

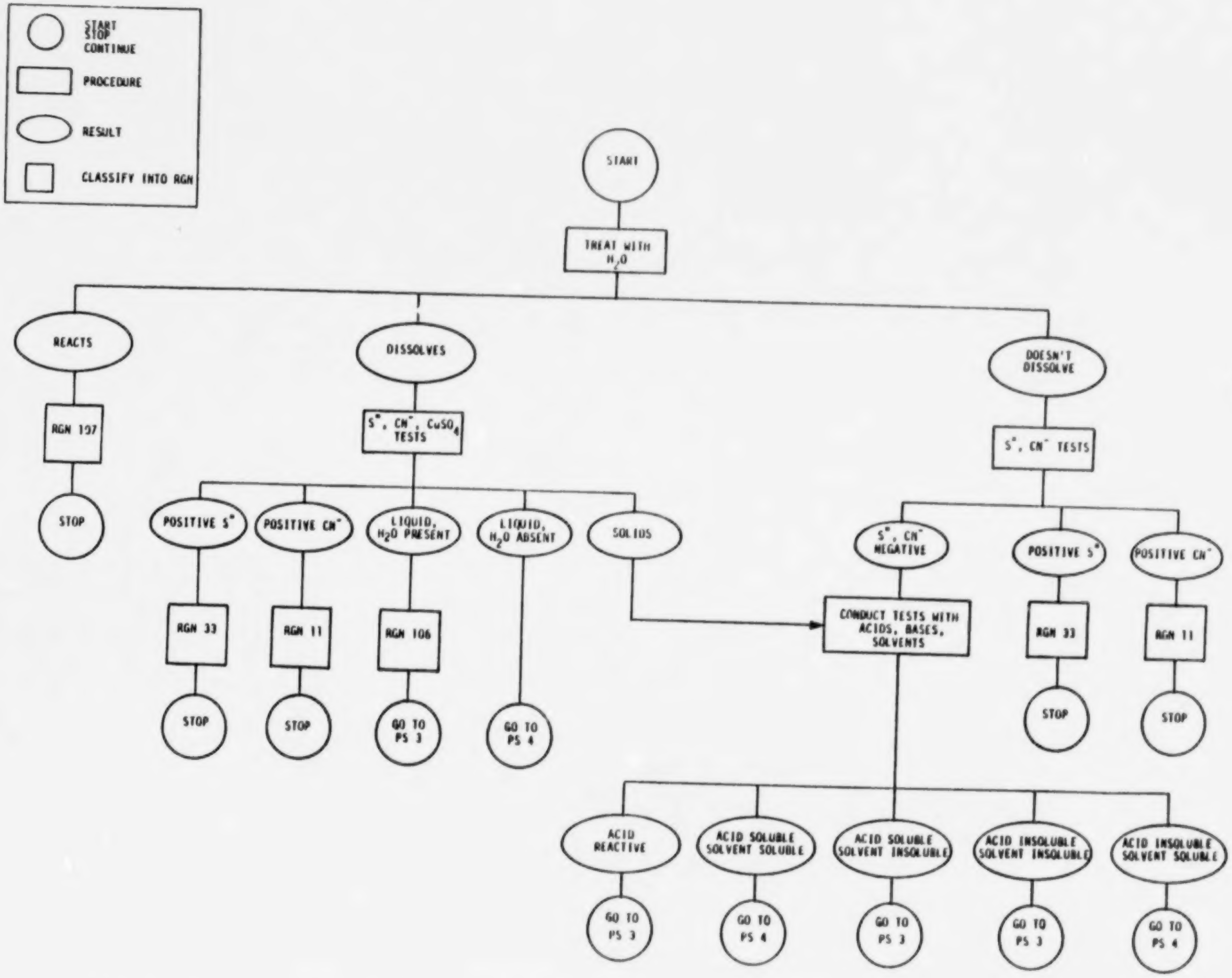

START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY INTO RGN
START
TREAT WITH H_2O
REACTS
RGN 197
STOP
DISSOLVES
$S^=$, CN^-, $CuSO_4$ TESTS
POSITIVE $S^=$
RGN 33
STOP
POSITIVE CN^-
RGN 11
STOP
LIQUID, H_2O PRESENT
RGN 106
GO TO PS 3
LIQUID, H_2O ABSENT
GO TO PS 4
SOLIDS
DOESN'T DISSOLVE
$S^=$, CN^- TESTS
$S^=$, CN^- NEGATIVE
CONDUCT TESTS WITH ACIDS, BASES, SOLVENTS
POSITIVE $S^=$
RGN 33
STOP
POSITIVE CN^-
RGN 11
STOP
ACID REACTIVE
GO TO PS 3
ACID SOLUBLE SOLVENT SOLUBLE
GO TO PS 4
ACID SOLUBLE SOLVENT INSOLUBLE
GO TO PS 3
ACID INSOLUBLE SOLVENT INSOLUBLE
GO TO PS 3
ACID INSOLUBLE SOLVENT SOLUBLE
GO TO PS 4

Figure A-3. Procedure set 2 -- solution/reactivity and special functionality tests.

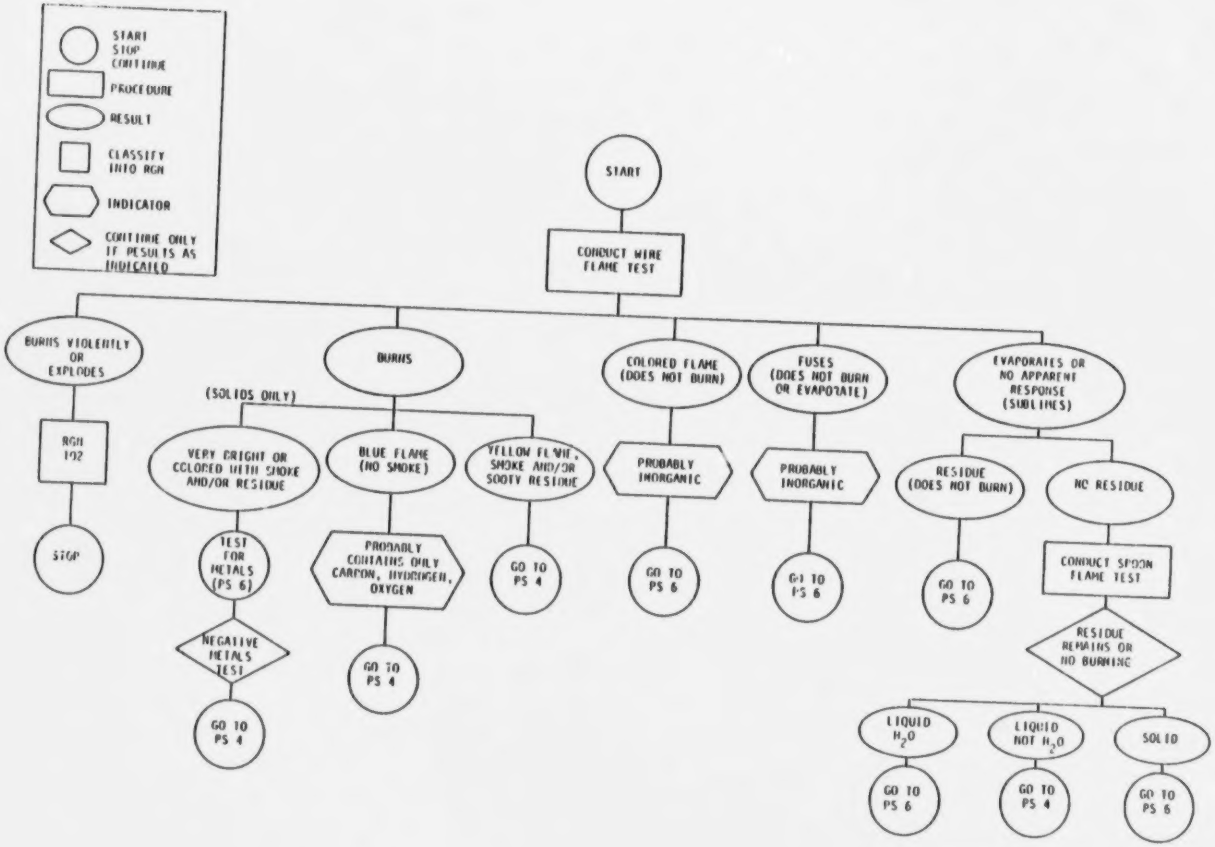

START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY INTO RGN
INDICATOR
CONTINUE ONLY IF RESULTS AS INDICATED
START
CONDUCT WIRE FLAME TEST
BURNS VIOLENTLY OR EXPLODES
RGN 102
STOP
BURNS
(SOLIDS ONLY)
VERY BRIGHT OR COLORED WITH SMOKE AND/OR RESIDUE
TEST FOR METALS (PS 6)
NEGATIVE METALS TEST
GO TO PS 4
BLUE FLAME (NO SMOKE)
PROBABLY CONTAINS ONLY CARBON, HYDROGEN, OXYGEN
GO TO PS 4
YELLOW FLAME, SMOKE AND/OR SOOTY RESIDUE
GO TO PS 4
COLORED FLAME (DOES NOT BURN)
PROBABLY INORGANIC
GO TO PS 6
FUSES (DOES NOT BURN OR EVAPORATE)
PROBABLY INORGANIC
GO TO PS 6
EVAPORATES OR NO APPARENT RESPONSE (SUBLIMES)
RESIDUE (DOES NOT BURN)
GO TO PS 6
NO RESIDUE
CONDUCT SPOON FLAME TEST
RESIDUE REMAINS OR NO BURNING
LIQUID H_2O
GO TO PS 6
LIQUID NOT H_2O
GO TO PS 4
SOLID
GO TO PS 6

Figure A-4. Procedure set 3 -- flame tests.

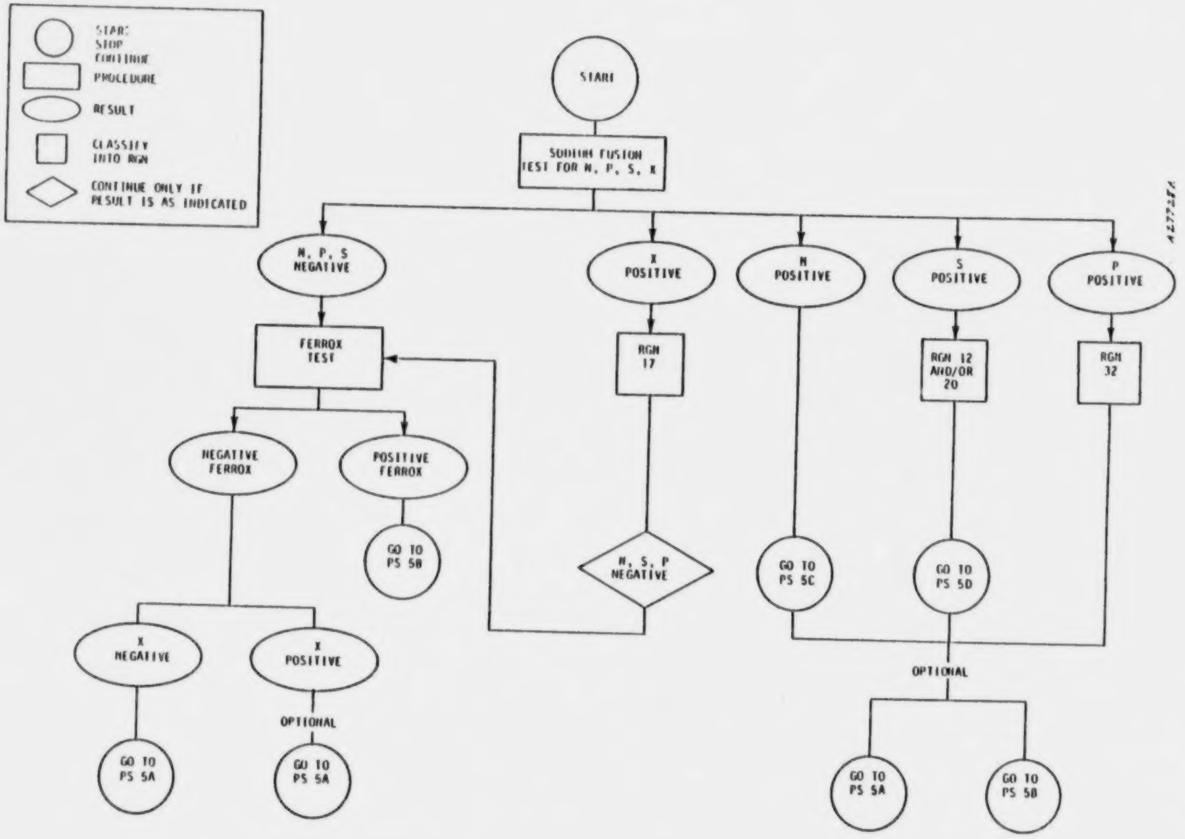

START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY INTO RGN
CONTINUE ONLY IF RESULT IS AS INDICATED
START
SODIUM FUSION TEST FOR N, P, S, X
N, P, S NEGATIVE
X POSITIVE
N POSITIVE
S POSITIVE
P POSITIVE
FERROX TEST
RGN 17
RGN 12 AND/OR 20
RGN 32
NEGATIVE FERROX
POSITIVE FERROX
GO TO PS 5B
N, S, P NEGATIVE
GO TO PS 5C
GO TO PS 5D
X NEGATIVE
X POSITIVE
OPTIONAL
OPTIONAL
GO TO PS 5A
GO TO PS 5A
GO TO PS 5A
GO TO PS 5B

Figure A-5. Procedure set 4 -- sodium fusion and ferrox tests.

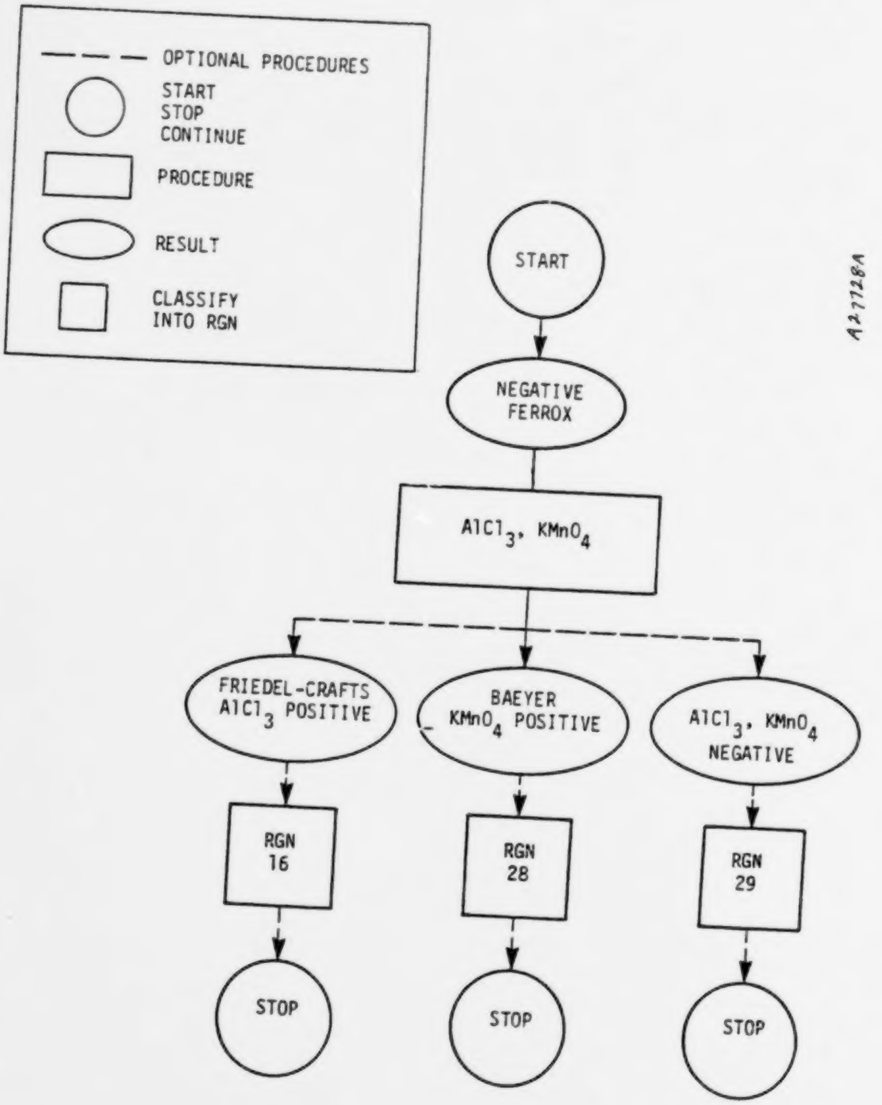

OPTIONAL PROCEDURES
START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY INTO RGN
START
NEGATIVE FERROX
$AlCl_3$, $KMnO_4$
FRIEDEL-CRAFTS $AlCl_3$ POSITIVE
BAEYER $KMnO_4$ POSITIVE
$AlCl_3$, $KMnO_4$ NEGATIVE
RGN 16
RGN 28
RGN 29
STOP
STOP
STOP
A27728A

Figure A-6a. Procedure set 5A -- organic functionality tests for hydrocarbon classification.

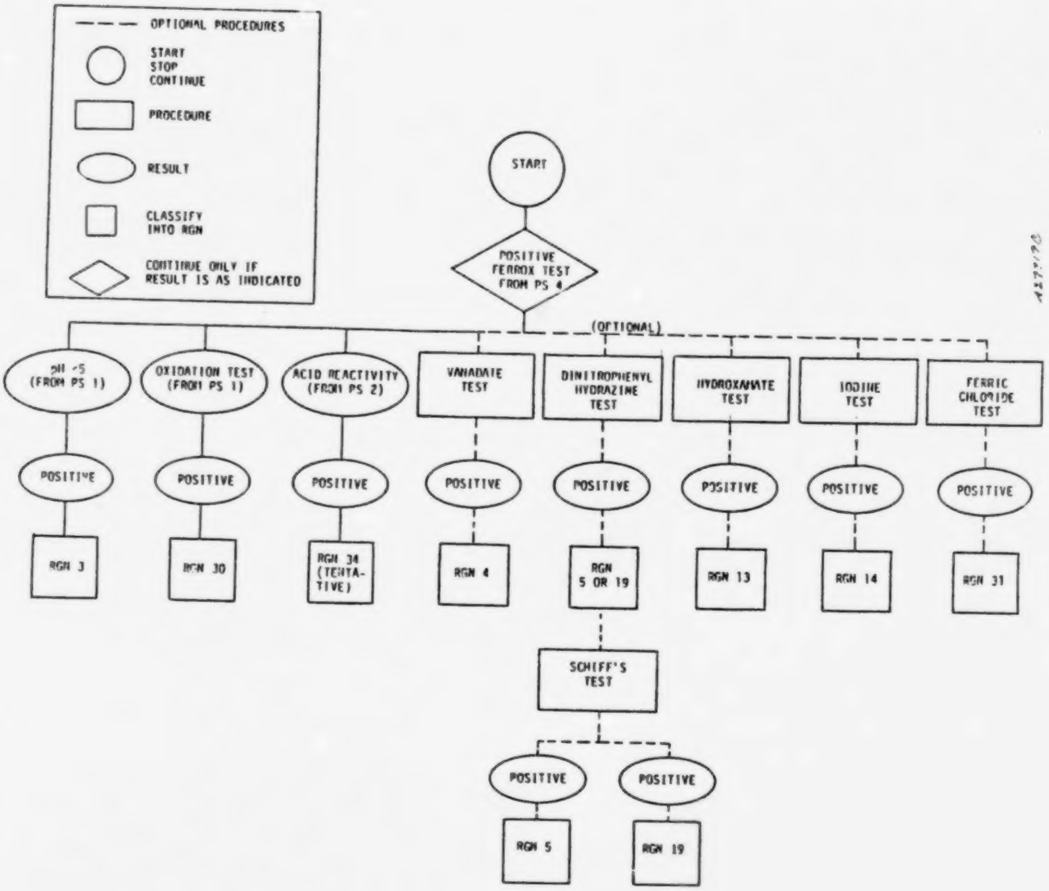
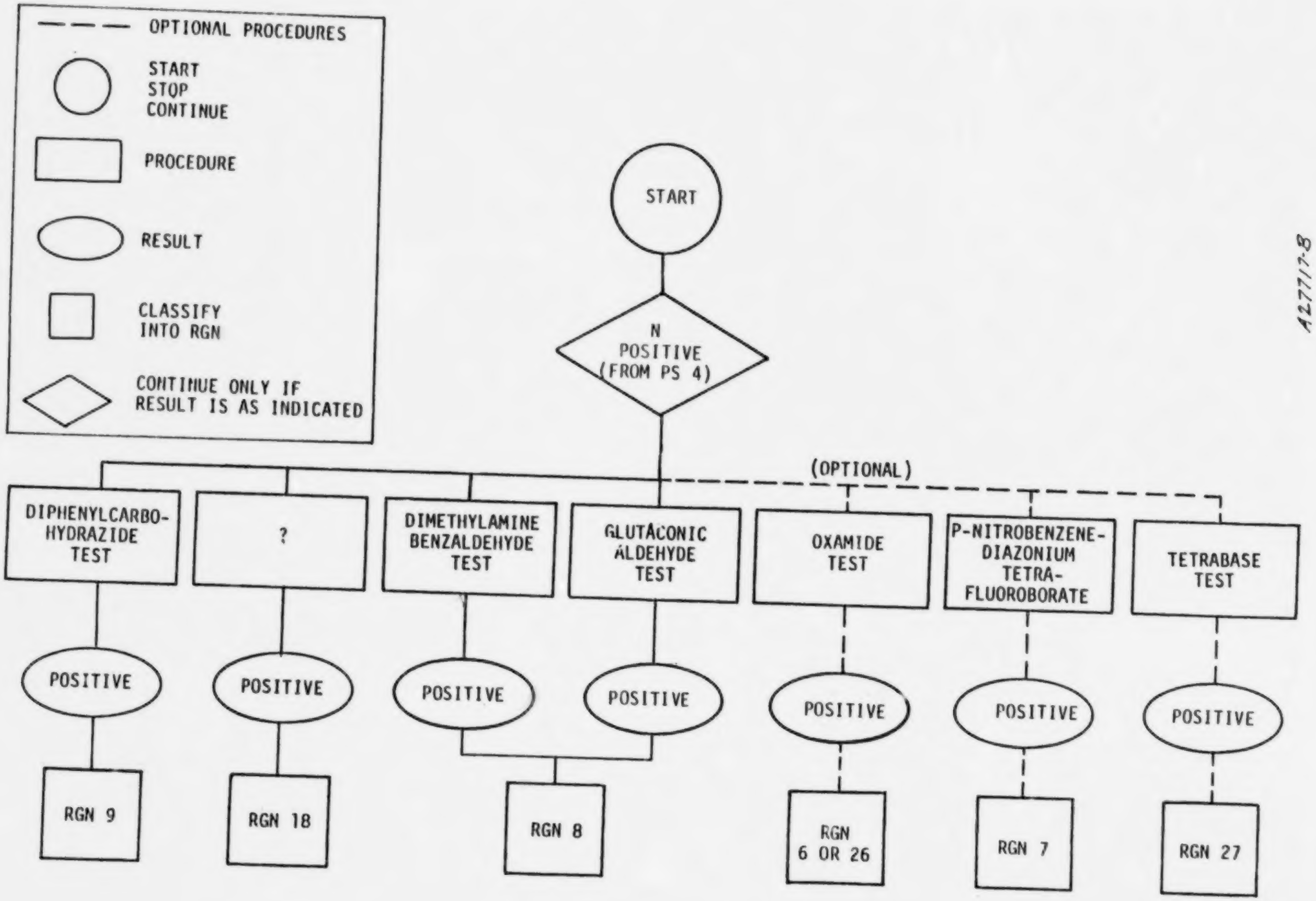
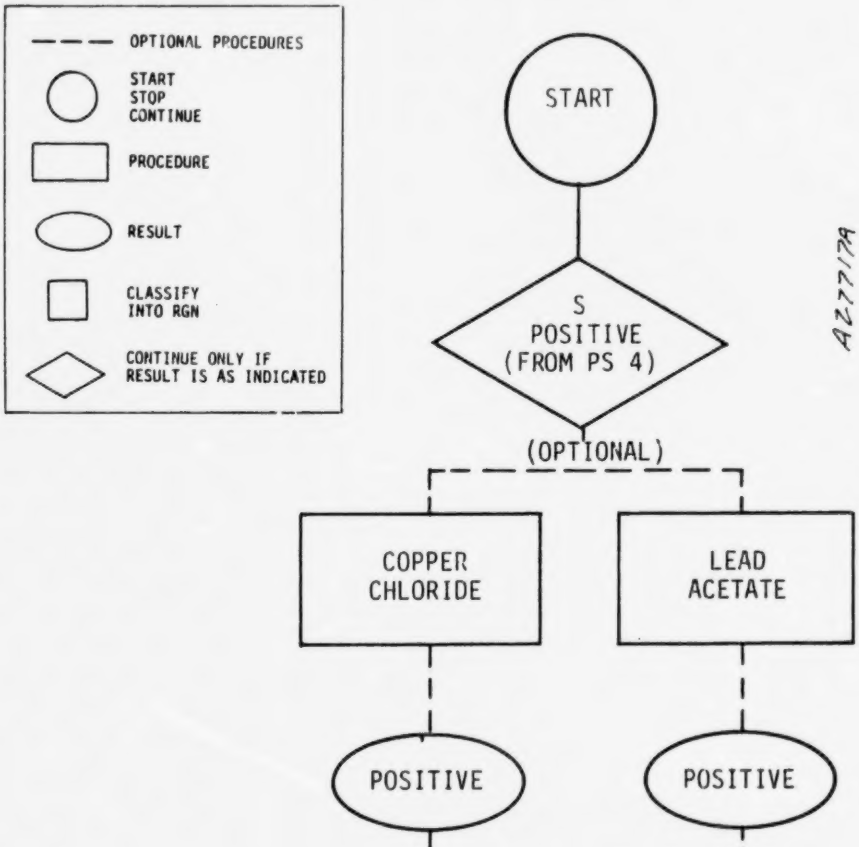

OPTIONAL PROCEDURES
START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY INTO RGN
CONTINUE ONLY IF RESULT IS AS INDICATED
START
POSITIVE FERROX TEST FROM PS 4
(OPTIONAL)
pH <5 (FROM PS 1)
OXIDATION TEST (FROM PS 1)
ACID REACTIVITY (FROM PS 2)
VANADATE TEST
DINITROPHENYL HYDRAZINE TEST
HYDROXAMATE TEST
IODINE TEST
FERRIC CHLORIDE TEST
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
RGN 3
RGN 30
RGN 34 (TENTATIVE)
RGN 4
RGN 5 OR 19
RGN 13
RGN 14
RGN 31
SCHIFF'S TEST
POSITIVE
POSITIVE
RGN 5
RGN 19

Figure A-6b. Procedure set 5B -- organic functionality tests for functional groups containing oxygen.


OPTIONAL PROCEDURES
START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY
INTO RGN
CONTINUE ONLY IF
RESULT IS AS INDICATED
START
N
POSITIVE
(FROM PS 4)
(OPTIONAL)
DIPHENYLCARBO-
HYDRAZIDE
TEST
?
DIMETHYLAMINE
BENZALDEHYDE
TEST
GLUTACONIC
ALDEHYDE
TEST
OXAMIDE
TEST
P-NITROBENZENE-
DIAZONIUM
TETRA-
FLUOROBORATE
TETRABASE
TEST
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
POSITIVE
RGN 9
RGN 18
RGN 8
RGN
6 OR 26
RGN 7
RGN 27

A27717-8

Figure A-6c. Procedure set 5C -- organic functionality tests for functional groups containing nitrogen.


OPTIONAL PROCEDURES
START
STOP
CONTINUE
PROCEDURE
RESULT
CLASSIFY INTO RGN
CONTINUE ONLY IF RESULT IS AS INDICATED
START
S POSITIVE (FROM PS 4)
(OPTIONAL)
COPPER CHLORIDE
LEAD ACETATE
POSITIVE
POSITIVE
RGN 12
RGN 20

A277179

Figure A-6d. Procedure set 5D -- organic functionality tests for functional groups containing sulfur.

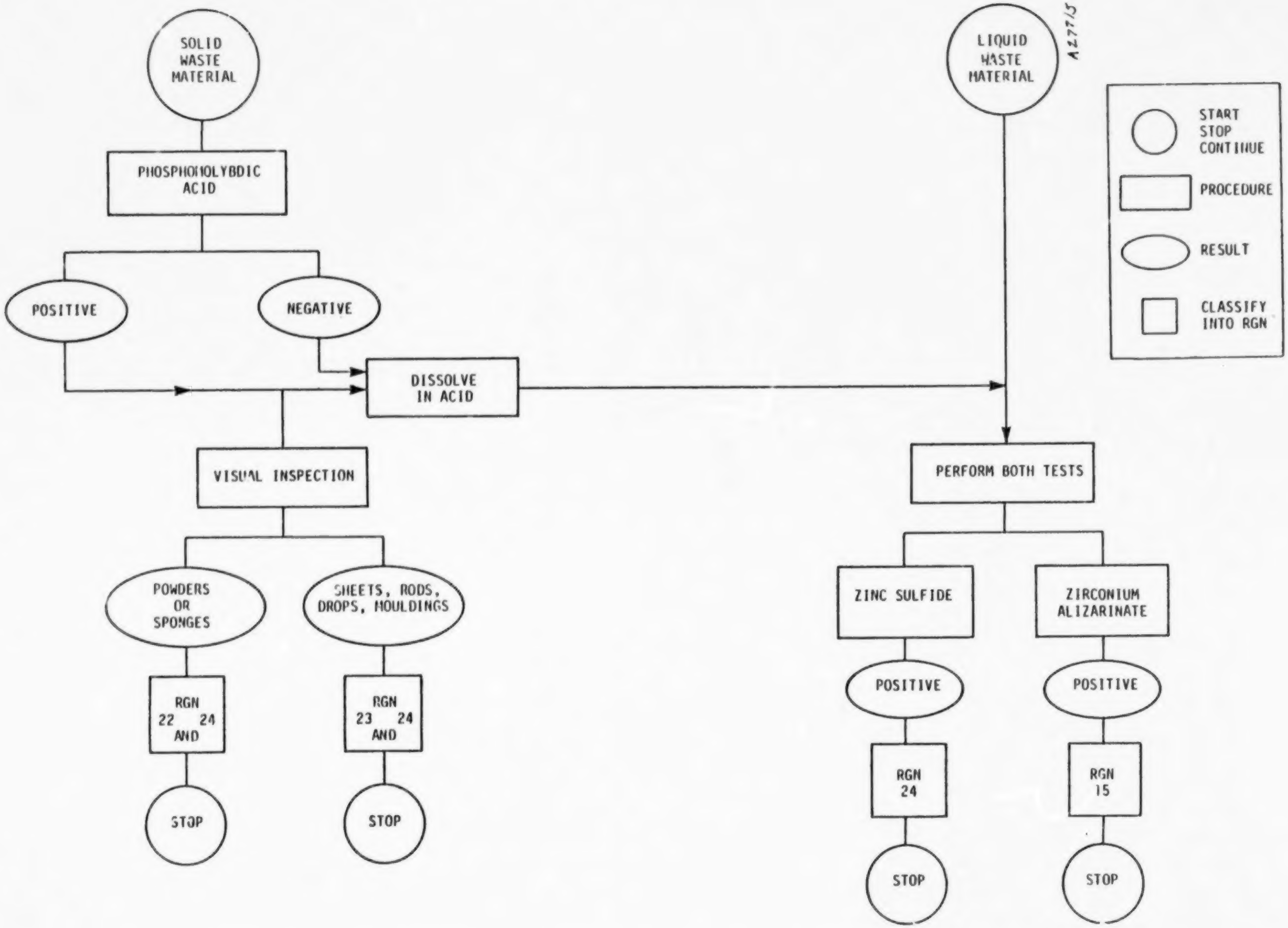

A27715
SOLID WASTE MATERIAL
PHOSPHOMOLYBDIC ACID
POSITIVE
NEGATIVE
DISSOLVE IN ACID
VISUAL INSPECTION
POWDERS OR SPONGES
SHEETS, RODS, DROPS, MOULDINGS
RGN 22 AND 24
RGN 23 AND 24
STOP
STOP
LIQUID WASTE MATERIAL
PERFORM BOTH TESTS
ZINC SULFIDE
ZIRCONIUM ALIZARINATE
POSITIVE
POSITIVE
RGN 24
RGN 15
STOP
STOP
START STOP CONTINUE
PROCEDURE
RESULT
CLASSIFY INTO RGN

Figure A-7. Procedure set 6 -- inorganic functionality tests.

fuming, bubbling, or spattering) are flagged and testing is suspended. Separate portions of the waste materials are then tested for sulfide (RGN 33) and cyanide (RGN 11); likewise, if these RGN's are present, further testing is suspended. Liquid waste materials are tested for the presence of water (RGN 106) with anhydrous copper sulfate. Acid, base, and solvent tests are performed (see Section A.4.2) except on materials containing sulfide or cyanide. Generally, materials that are soluble in organic solvents are classified as organic, and the testing is continued with PS 4. Sulfide and cyanide containing wastes are considered inorganic.

CAUTION

Sulfide and cyanide wastes must not be treated with acid.

Materials that are soluble in water and acids but not in solvents, or are insoluble in water, acid, and solvents may be organic or inorganic, and testing is continued with PS 3.

A.2.2.3 PS 3 -- Flame Test--

Observation of the behavior of a material upon ignition can provide a great deal of insight regarding its composition. Results (observations) upon ignition are characterized by one of the following descriptions:

- Burns violently
- Burns (with or without smoke)
- Produces a colored flame, but does not burn
- Fuses (melts) but does not burn
- Evaporates or sublimes but does not burn

Figure A-4 shows the classification observations. While these observations are somewhat subjective, it is anticipated that most observers can make a distinction between organics, inorganics, and free metals using the flame test. RGN 107 (explosive) is determined directly by means of the flame test.

In summary, materials that burn are classificed as organic. Materials that melt, evaporate, or only color a flame are classified as inorganic.

In addition, several observations are made that can give further clues regarding the composition of the material. A material that burns with a soft blue flame (no smoke) is most likely an organic material containing only carbon, hydrogen, and oxygen. Materials that burn with a yellow flame and produce a sooty flame are probably aromatic or contain nitrogen, sulfur, or phosphorus.

The test procedures are explained in Section A.4.3.

CAUTION

> It is always advisable to begin testing with a small portion to safely identify materials that are unusually flammable or explosive.

If explosive materials are encountered, it is highly advisable to suspend further testing at this point.

It should be noted that this procedure generally does not identify the potential of water-based organics. If, after conducting PS 6, no RGN's are identified except RGN 106 (water and mixtures containing water), it is suggested that the waste be considered as water-containing organics. Some organic functionality tests can be performed; however the sodium fusion procedure should not be performed with materials containing water as the major constituent.

A.2.2.4 PS 4 -- Sodium Fusion and Ferrox Tests--

If it is determined, by results of PS 2 and/or PS 3 that a waste material is organic, the sodium fusion test is performed to determine the presence of halogen (chlorine, bromine, or iodine) (X), sulfur (S), nitrogen (N), and/or phosphorus (P). The ferrox test is performed, in the absence of sulfur, nitrogen, or phosphorus, to determine the presence of oxygen (O). The flow diagram for PS 4 is shown in Figure A-5.

Note

> The sodium fusion procedure is not recommended for explosive materials.

If the waste material is found to contain nitrogen, sulfur, or phosphorus, testing is continued with PS 5 C, D, or E as appropriate. If any of these elements is present, oxygen is also assumed to be present, and PS 5B is conducted. PS 5B is also conducted if the result of the ferrox test is positive. If the ferrox and halogen test results are negative, optional testing is continued to classify hydrocarbons (PS 5A). If, on the other hand, the ferrox test is negative and halogen is present, the testing sequence is complete. If the halogen test is positive, the analyst may wish to conduct PS 5A to differentiate between halogenated aromatics (e.g., PCB's), halogenated alkanes (e.g., chloroform), and halogenated alkenes (e.g., trichloroethylene).

In summary, PS 4 is used to determine the elements present in an organic waste material. The results are used as a guide for which functionality tests are to be performed in PS 5. The test procedures are outlined in Section A.4.4.

A.2.2.5 PS 5 -- Organic Functionality Tests--

Elemental analysis information obtained in PS 4 is used as a starting point for PS 5. This set of procedures includes specific tests for 21 RGN's, and is outlined in Figures A-6a through A-6d. PS 5 contains four major subsets: tests for hydrocarbon classification and tests for functionalities containing sulfur, nitrogen, and/or oxygen only.

The 21 reactivity groups determined in PS 5, arranged according to procedure subset, are listed in Table A-4.

Each of the procedure subsets 5B through 5D, is performed only if the respective element was determined to be present in PS 4. If the element was found to be absent in PS 4, it can be concluded that all reactivity groups determined in that subset are absent. If sulfur, nitrogen, or phosphorus are present, oxygen is also assumed to be present, and PS 5B is conducted in addition to the appropriate subset. If oxygen, nitrogen, halogen, sulfur, and phosphorus are all found to be absent, it is concluded that the material is hydrocarbon and PS 5A is conducted. If any of these listed elements are present, the three hydrocarbon RGN's are considered to be absent. Each of the individual procedure subsets are explained in the following paragraphs.

A.2.2.5.1 PS 5A -- hydrocarbon classification tests (optional)--Three classes of hydrocarbons (RGN's 16, 28, and 29) are distinguished by the Friedel-Crafts test and the Baeyer test. The flow diagram for PS 5A is shown in Figure A-6a. These three RGN's are mutually exclusive (by definition), therefore, if one test result is positive, the other need not be run. It is suggested that the Friedel-Crafts test be performed first in this sequence. If both the Friedel-Crafts test and the Baeyer test are negative, the material is classified as RGN 29.

Since the hydrocarbon RGN's are similar in their compatibility characteristics and are all relatively unreactive, this procedure subset is labeled "optional." The test procedures for these tests are outlined in Section A.4.5.1.

It is frequently desirable to detect aromatics, even in the presence of functional groups. It is therefore recommended that the Friedel-Crafts test be performed, particularly if organic halogens are present.

A.2.2.5.2 PS 5B -- tests for organic functional groups containing oxygen--Tests for functional groups containing oxygen involve tests for nine specific RGN's. These tests are performed on all organic wastes except those materials shown not to contain sulfur, nitrogen, or phosphorus by the fusion procedure and those that give a negative ferrox test. The flow diagram for procedure subset 5B is shown in Figure A-6b and the test procedures are presented in Sections A.4.5.2.1 through A.4.5.2.7.

Three of the RGN's are identified by means of results obtained in PS 1 and PS 2. Organic acids (RGN 3) are identified by means of their pH (pH 2 to 5). Organic peroxides (RGN 30) respond as oxidizing agents and should be identified in PS 2. The result is confirmed by a special paper test

TABLE A-4. RGN'S DETERMINED IN PS 5

- PS 5A -- Hydrocarbons
  - RGN 16 -- Aromatic hydrocarbons
  - RGN 28 -- Unsaturated aliphatic hydrocarbons
  - RGN 29 -- Saturated aliphatic hydrocarbons
- PS 5B -- Oxygen functional groups
  - RGN 3 -- Organic acids
  - RGN 4 -- Alcohols and glycols
  - RGN 5 -- Aldehydes
  - RGN 13 -- Esters
  - RGN 14 -- Ethers
  - RGN 19 -- Ketones
  - RGN 31 -- Phenols and cresols
  - RGN 34 -- Epoxides
  - RGN 30 -- Peroxides
- PS 5C -- Nitrogen functional groups
  - RGN 6 -- Amides
  - RGN 7 -- Amines
  - RGN 8 -- Azo compounds, diazo compounds, and hydrazines
  - RGN 9 -- Carbamates
  - RGN 18 -- Isocyanates
  - RGN 26 -- Nitriles
  - RGN 27 -- Organic nitro compounds
- PS 5D -- Sulfur functional groups
  - RGN 12 -- Dithiocarbamates
  - RGN 20 -- Mercaptans and other organic sulfides

(Section A.4.5.1.1). No specific test is included for epoxides (RGN 34), but this RGN is tentatively identified by its vigorous, sometimes violent reaction with mineral acids, particularly nitric acid.

The remaining six RGN's identified in PS 5B are less reactive than RGN's 3, 30, or 34. If one of these reactive RGN's are identified, it is advisable to omit further tests for other RGN's. Furthermore, compatibility characteristics of the less reactive RGN's are similar, so tests for the remaining six RGN's are labeled optional.

Dinitrophenylhydrazine is used to identify carbonyl compounds (aldehydes and ketones, RGN's 5 and 19), and the Schiff's test is used to distinguish between the two. Separate tests are conducted to identify alcohols (RGN 4), esters (RGN 13), ethers (RGN 14), and phenols (RGN 3).

A.2.2.5.3 PS 5C -- tests for organic functional groups containing nitrogen--Functional group tests for organic compounds containing nitrogen are contained in PS 5C. These tests are performed only if nitrogen is detected by the sodium fusion procedure (PS 4). The flow diagram for classification of the seven RGN's containing nitrogen is shown in Figure A-6c. The test procedures are described in Sections A.4.5.3.1 through A.4.5.3.5.

Three RGN's are considerably more reactive than others: carbamates (RGN 9), isocyanates (RGN 18), and azo compounds, diazo compounds, and hydrazines (RGN 8). The diphenylcarbohydrazide test is conducted to identify RGN 9. Azo compounds are identified by the dimethylaminobenzaldehyde test and hydrazines are identified by the glutaconic aldehyde test. If either of these tests are positive, the material is classified as RGN 8. No suitable test procedure is included for isocyanates (RGN 18).

If a material is classified as RGN 8, 9, or 18, it is suggested that further testing in PS 5C be eliminated. Tests for RGN's 6, 7, 26, and 27 are included, but are labeled optional, because compatibility characteristics of these materials are similar and the reactivity hazard of these materials is not as great. Amides and nitriles are not distinguished by the test procedure, but these materials are very similar in their reactivity properties.

A.2.2.5.4 PS 5D -- tests for organic functional groups containing sulfur--Tests for two RGN's that contain sulfur are included in PS 5D. Since the reactivity characteristics of these RGN's are similar, the procedure subset is labeled optional. If sulfur is shown to be absent in the sodium fusion procedure (PS 4), both RGN's 12 and 20 are absent.

The flow diagram for procedure subset 5D is shown in Figure A-6d, and the test procedures for RGN's 12 and 20 are outlined in Sections A.4.5.4.1 through A.4.5.4.2.

A.2.2.6 PS 6 -- Inorganic Functionality Tests--

If a waste material is classified as inorganic in PS 1-3, three test procedures are conducted to identify inorganic materials not already identified (PS 1 and 2). The flow diagram for PS 6 is shown in Figure A-7 and specific test procedures are described in Sections A.4.6.1 to A.4.6.2.

Elemental metals or alloys can often be identified visually; their presence is confirmed by the phosphomolybdic acid test. Alkali and alkaline earth metals (RGN 21) react with water to give solutions that are basic (PS 2). Visual inspection can be used to identify the form of elemental metals (RGN's 22 or 23). It should be noted that all elemental metals except mercury (which is readily identified visually) are solids, so the phosphomolybdic acid test is not conducted if the waste material under investigation is a liquid.

Toxic metal compounds (RGN 24) are identified by a simple zinc sulfide test, and inorganic fluorides (RGN 15) are identified by the zirconium alizarinate test. No specific test procedure is included for nitrides (RGN 25) but these materials are explosive and are thus detected in PS 3.

A.3 HAZARDOUS WASTE CLASSIFICATION FIELD DATA SHEET

A data sheet used in recording the results of hazardous waste classification tests is shown in Figure A-8. As procedure sets are completed the appropriate sections are filled in. Conclusions regarding the classification of materials into reactivity groups are recorded at the bottom of the data sheet. RGN's identified during the course of the testing sequence are circled. Numbers are crossed out for those RGN's found to be absent. At the end of the test sequence, the circled reactivity groups are used to define the gross chemical composition and thus determine compatibility characteristics of the waste material.

Note

It is important to consult Sections A.2 and A.4 of the test manual for the proper interpretation of test results.

A.4 TEST PROCEDURES

A.4.1 PS 1 -- pH and Redox Tests

A.4.1.1 pH Tests for Acids and Caustics (RGN's 1, 2, 3, 10)--

Procedure:

The pH is determined by spotting the liquid material or the solid suspended or dissolved in water on the pH paper and comparing the color with the corresponding color and pH from the color table. The test paper is moistened with water before applying the test material.

Substances that do not dissolve in water or interfere with the color reactions of the pH paper cannot be tested for pH with this procedure.

Sample: ____________________ Analyst: ____________________

____________________ Date: ____________________

Preliminary Observations: ____________________

____

____

____

PROCEDURE SET 1

pH __________ (RGN 1, 2, 3, 10)

Oxidation __________ (RGN 104) Reduction __________ (RGN 105)

PROCEDURE SET 2

H_2O React __________ (RGN 107) H_2O Present __________ (RGN 100)

S __________ (RGN 33) CN __________ (RGN 11)

Solubility-Reactivity Tests

H_2O ____________________

HCl ____________________

HNO_3 ____________________

H_2SO_4 ____________________

NaOH ____________________

Methanol ____________________

Acetone ____________________

Toluene ____________________

Hexane ____________________

PROCEDURE SET 3

Burns Violently ____________________ (RGN 102)

Observations: ____________________

____

____

PROCEDURE SET 4

Sodium Fusion: N __________ S __________ SCN __________

P __________ (RGN 32) X __________ (RGN 17)

Ferrox __________

Figure A-8. Hazardous waste classification field test results (page 1 of 2).

Sample: ______________________________
Date: ______________________________

PROCEDURE SET 5

PS 5A Hydrocarbons
$AlCl_3$ __________ (RGN 16) $KMnO_4$ __________ (RGN 28) __________ (RGN 29)

PS 5B Oxygen RGN's
pH __________ (RGN 3) Oxidation _________ (RGN 30)
__________ (RGN 34) Vanadate __________ (RGN 4, 31)

DNPH __________ (RGN 5 or 19) Schiff's __________ (RGN 5)
Hydroxamate __________ (RGN 13) Iodine __________ (RGN 14)
Ferric Chloride __________ (RGN 31)

PS 5C Nitrogen RGN's
DNPCH __________ (RGN 9) __________ (RGN 18)
DMAB __________ Glut. Ald __________ (RGN 8)
Oxamide __________ (6 or 26) DBTFB __________ (RGN 7)
Tetrabase __________ (RGN 27)

PS 5D Sulfur RGN's
Copper Chloride __________ (RGN 12)
Lead Acetate __________ (RGN 20)

PROCEDURE SET 6

Phosphomolybdic Acid __________ (RGN 21, 22, or 23)
ZnS __________ (RGN 24)
Zirconium Alizarinate __________ (RGN 15)

REACTIVITY CLASS(ES) (circle)

1	5	9	13	17	21	25	29	33	102	106
2	6	10	14	18	22	26	30	34	103	107
3	7	11	15	19	23	27	31	35	104	
4	8	12	16	20	24	28	32	36	105	

Comments: ____

____

____

____

____

Figure A-8. Hazardous waste compatibility field test results (page 2 of 2).

A.4.1.2 Redox Tests (RGN's 104 and 105)--

Procedure:

To test for reducing agents filter paper strips impregnated with methylene-blue and/or with 2,6-dichloroindophenol are spotted with the liquid or dissolved test material. Strong reducing agents decolorize both methylene blue and 2,6-dichlorcindophenol. Weak reducing agents decolorize 2,6-dichloroindophenol only, which turns pale pink and then colorless.

Oxidizing agents are tested by spotting the liquid or dissolved test material on potassium-iodide starch paper which turns purple when an oxidizing agent is present.

Reagents:

1. Methylene blue: 0.2g of methylene blue are dissolved in 100 mL of water.
2. 2,6-dichloroindophenol: 0.2g of 2,6-dichloroindophenol are dissolved in 100 mL of ethanol.
3. KI/starch paper: Potassium-iodide starch paper (VWR Scientific)
4. Filter paper strips: Whatman filter paper disks were impregnated with the indicator solutions, dried, cut into strips and stored in closed glass vials. The papers can be used for at least 3 months.

A.4.2 PS 2 -- Solution-Reactivity and Special Functionality Tests

A.4.2.1 Solution-Reactivity Tests--

Procedure:

The reactivity of the compounds is tested with water, concentrated nitric acid, concentrated sulfuric acid, 3M sodium hydroxide, hexane, toluene, acetone, and methanol. The test material is placed on a ceramic spotplate -- 2 drops or a small spatula tip per cavity for each reagent. Then 1 to 3 drops of the reagents are added and the reactions are recorded. The reaction characteristics recorded are: solubility/miscibility, coloration, gas evolution, fuming, charring, precipitation, violence of reaction. In cases where the miscibility or formation of a precipitate is difficult to observe, the reaction mixture is drawn into a Pasteur pipet for better observation.

A.4.2.2 Cyanide Test--

Procedure:

Cyanide is determined by treating 2 to 3 drops of the liquid waste solution in water with 1 drop of chloramine T solution followed by 1 drop of pyridine-barbituric acid solution. If the solution turns a dark red or carmine after 10 to 30 sec, it is a positive response.

Reagents:

1. Chloramine T solution: 1g of chloramine T is dissolved in 100 mL of distilled water.

2. Pyridine-barbituric acid: 1.5g of barbituric acid (Aldrich Chemical Company) is mixed with 5 mL of water and 7.5 mL of pyridine. The mixture is treated with 1.5 mL of hydrochloric acid (concentrated), and diluted to 25 mL.

A.4.2.3 Sulfide Test--

Procedure:

The presence of sulfide is determined by placing a strip of lead-acetate paper moistened with 1 drop of water over the spotplate cavity containing 2 to 3 drops of the waste solution and 1 to 3 drops of 3M hydrochloric acid. Black PbS forms on the paper after 0.5 to 1 min if sulfide is present.

Reagents:

1. 3M HCL: 25 mL of concentrated hydrochloric acid is diluted to 100 mL with distilled water.

2. Lead-acetate paper: (VWR Scientific)

A.4.2.4 Copper Sulfate Test for Water--

Liquid waste materials are tested for the presence of water with anhydrous copper sulfate. A small spatula tip of anhydrous copper sulfate is treated with a few drops of waste material in the depression of a porcelain spotplate. The copper sulfate turns dark blue in the presence of water.

Reagent:

1. Copper sulfate: anhydrous copper sulfate ($CuSO_4$) is used.

A.4.3 PS 3 -- Flame Tests

Procedure:

Depending on the type and intensity of the reaction, the flame test is carried out with a wire loop, a spatula, or with a ceramic spoon.

Wire Loop. The wire loop is dipped in concentrated hydrochloric acid and heated until the flame regains its original color. After dipping the wire in distilled water, a few crystals or drops of the compound are scooped up and kept just above the flame for a few seconds. Then it is lowered into the hot part of the flame and the reactions such as color of flame, smoke, sparking, and residue are observed and recorded.

Spatula. To observe residues from inorganic salts, or when the observations with the wire loop are inconclusive, it becomes necessary to use more material. The spatula tip is cleaned in the same manner as the wire loop and a spatula-tip full of material is heated.

Ceramic Spoon. A good way to test for residues, especially from solutions of inorganics or liquids, is to heat the material on a ceramic spoon.

Note:

1. It is important to start out with a small amount on the wire loop in case of a violent reaction of the test material with the flame.

A.4.4 PS 4 -- Sodium Fusion and Ferrox Test

A.4.4.1 Sodium Fusion Procedure--

Procedure:

A spatula tip of the solid test material or a few drops of the liquid test material is placed in a 4-in. Pyrex test tube. A pellet of sodium is added and the mixture is carefully heated to a red glow. In some cases a light flash can be observed when the sodium reacts with the test material. When the mixture is still red-hot, more test material is added and the mixture is again heated to a red glow. After cooling, methanol is added dropwise to destroy the excess sodium. After all bubbling has ceased about 2 mL of distilled water is added and the mixture is filtered. The filtrate is then tested for cyanide, sulfide, halide, and phosphate according to procedures A.4.4.1.1 through A.4.4.1.4. The tests can be performed in a ceramic spotplate or by transferring small portions of the test mixture to small test tubes.

Reagents:

1. Sodium pellets in mineral oil.
2. Methanol.

CAUTION

A few classes of organic compounds such as nitroalkanes, organic azides, diazo esters, diazonium salts, and some aliphatic polyhalides (chloroform, carbon tetrachloride) react explosively with hot sodium or magnesium. Safety goggles always should be worn when these decompositions are being carried out.

Notes:

1. This test must be carried out in a well-ventilated area behind a safety shield.
2. It is recommended that explosive materials not be subjected to the sodium fusion procedure.

A.4.4.1.1 Cyanide test for nitrogen (N)--

Procedure:

Nitrogen is determined by treating 2 to 3 drops of the fusion extract with 1 drop of chloramine T solution followed by 1 drop of pyridine-barbituric acid solution. If the mixture turns a dark red or carmine after 10 to 30 sec, it is a positive response.

Reagents:

1. Chloramine T solution: 1g of chloramine T is dissolved in 100 mL of distilled water.
2. Pyridine-barbituric acid: 1.5g of barbituric acid (Aldrich Chemical Company) is mixed with 5 mL of water and 7.5 mL of pyridine. The mixture is treated with 1.5 mL of hydrochloric acid (concentrated), and diluted to 25 mL.
3. Ferric chloride: 13.5g of ferric chloride ($FeCl_3$) is dissolved in 100 mL of distilled water and 2 mL of concentrated hydrochloric acid.

Note:

1. Some organic materials containing both nitrogen and sulfur form thiocyanate and give a negative cyanide test. Therefore, if a negative cyanide test and a positive sulfide test are obtained, the fusion extract is tested for thiocyanate. Two drops of fusion extract are treated with one drop of dilute hydrochloric acid and one drop of ferric chloride. Red indicates a positive test.

A.4.4.1.2 Sulfide test for sulfur (S)--

Procedure:

The presence of sulfide is determined by placing a strip of lead-acetate paper moistened with 1 drop of water over the spotplate cavity containing 2 to 3 drops of the test solution and 1 to 3 drops of 3M hydrochloric acid. The test paper turns black after 0.5 to 1 min if sulfur is present.

Reagents:

1. 3M HCL: 25 mL of concentrated hydrochloric acid is diluted to 100 mL with distilled water.
2. Lead-acetate paper: (VWR Scientific)

A.4.4.1.3 Silver nitrate test for halogen (X)--

Procedure:

To the acidified (1 to 2 drops nitric acid) filtrate a few drops of silver nitrate solution are added. A precipitate indicates the presence of

Cl^-(white), Br^-(light brown), or I^-(pale yellow). If sulfide or cyanide is present in the fusion extract it is removed by boiling 1 mL of the fusion extract with a few drops of 3M HNO_3 before carrying out the halogen test.

Reagents:

1. Silver nitrate solution: 1.69g of silver nitrate dissolved in 100 mL of distilled water.

2. 3M HNO_3

A.4.4.1.4 Phosphate test for phosphorus (P)--
Procedure:

To 2 to 3 drops of the test solution, 2 drops of ammonium molybdate solution are added. A bright yellow precipitate indicates the presence of phosphate. The test result might be obscured when the fusion filtrate is colored.

Reagent:

1. Ammonium molybdate solution: 6g of ammonium molybdate, $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ (J. T. Baker Chemical Company) are dissolved in 150 mL of water and 35 mL of concentrated hydrochloric acid.

A.4.4.1.5 Alternate phosphate test for phosphorus--
Procedure:

To about 2 mL of the test solution, 8 drops of molybdate solution are added in a test tube. After addition of 1 drop of stannous chloride the mixture turns deep blue after 10 to 30 sec in the presence of phosphate. It is very important to use the reagents in the correct proportions and order described.

Reagents:

1. Ammonium molybdate/H_2SO_4: 5g of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ are dissolved in 35 mL of H_2O. Then 15.5 mL of concentrated H_2SO_4 is added and the solution is diluted with water to 200 mL after cooling.

2. Stannous chloride: 1.0g of stannous chloride $\cdot$ $2H_2O$ is dissolved in 40 mL of warmed (approximately 60°C in a hot water bath) glycerol.

A.4.4.2 Ferrox Test For Organic Compounds Containing Oxygen--
Procedure:

A glass stirring rod is dipped into the ether solution of the reagent to a depth of about 0.5 cm. The ether is allowed to evaporate in to the air. A few drops of the test solution or an equivalent amount of solid with a few drops of methylene chloride, chloroform, or hexane are placed in the depression of a spotplate. The sample is stirred with the reagent-coated stirring rod.

A positive test is indicated if the test solution turns red. Occasionally, some red scratches will appear on the spotplate with a negative test. (It is advisable to test a small portion of the solvent alone as a control.)

Reagent:

1. Ferric hexathiocyanatoferriate (ferrox): A solution of 4g of potassium thiocyanate (KCNS) in 20 mL of water is mixed with a solution of 5g of ferric chloride ($FeCl_3 \cdot 6H_2O$) in 20 mL of water. The solution is extracted with 30 mL of ether. The ether (upper) layer is stored in a dark container prior to use, and has been found to be stable for several weeks when stored at room temperature.

Notes:

1. Negative tests were obtained for some compounds containing oxygen bound to nitrogen (i.e., picrates)
2. A positive test that fades was obtained for some easily oxidized compounds (i.e., mercaptans)

A.4.5 PS 5 -- Organic Functionality Tests

A.4.5.1 PS 5A -- Hydrocarbon Classification Tests--

A.4.5.1.1 Friedel-Crafts test for aromatic hydrocarbons (RGN 16) --
Procedure:

A small amount of anhydrous aluminum chloride is placed in a 4-in. test tube and heated with a small hot flame to sublime the $AlCl_3$ on the test tube wall. A small amount of the test material is dissolved in 0.5 mL $CHCl_3$ and a few drops of this solution is run down the side of the test tube. On contact with the sublimed $AlCl_3$, aromatic hydrocarbons form brightly colored products, which range from yellow, orange or red, to blue.

Reagents:

1. Aluminum chloride: anhydrous powder (Aldrich Chemical Company)
2. Chloroform

Notes:

1. A sufficiently large amount of $AlCl_3$ should be used to cover a 1 to 2 cm^2 area of the test tube wall with sublimed $AlCl_3$ to make the color formation easily visible.

2. It is often desirable to determine the presence of the aromatic structure in the presence of other functional groups (especially chloride). This test can be performed in the presence of many functional groups with little interference.
3. The specific color produced in the test can often be used as an indicator of the type of aromatic structure present. Some general guidelines are as follows:

Color	Structure indicated
Yellow, orange, red	Monocyclic (benzene)
Blue	Bicyclic (naphthalene)
Green	Complex aromatic (anthracene)

A.4.5.1.2 Baeyer (Permanganate) test for unsaturated aliphatic hydrocarbons (RGN 28)--

Procedure:

Five drops of a small amount of the test material in acetone are added to 2 to 3 drops of $KMnO_4$ reagent. If the test material contains unsaturated hydrocarbons, the reagent is decolorized (with formation of brown MnO_2). The reaction occurs instantaneously or after 0.5 to 1 min of stirring.

Reagents:

1. $KMnO_4$ reagent: 0.5g $KMnO_4$ in 50 mL of acetone
2. Acetone

Note:

1. It is advisable to prepare the reagent immediately prior to use. This is most conveniently accomplished by placing 0.5g of $KMnO_4$ in an empty dropping bottle and adding acetone upon arrival at the test site.

A.4.5.2 PS 5B -- Tests for Functional Groups Containing Oxygen--

A.4.5.2.1 Peroxide Test (RGN 30)--

Procedure:

One drop of the liquid test substance or a few crystals of the test material dissolved in water, alcohol, or acetone is placed on a peroxide test strip. The development of a blue color indicates the presence of peroxides, the color intensity is an indicator of the peroxide concentration.

Reagent:

1. Peroxide test strip EM Quant (Merck)

Notes:

1. Oxidizing ions can interfere but they turn the test strip grey to brown instead of the characteristic blue.

2. The color development requires the presence of traces of water, which can be achieved by adding a drop of water to the nonaqueous test solution or by exposing the strip to humid air for 1 to 3 min.

A.4.5.2.2 Vanadate test for alcohols (RGN 4)--

Procedure:

A small amount of the solid test material or 1 drop of the liquid test material are placed in a test tube with 1 to 2 drops of toluene. Five drops of the vanadate-quinolinol reagent are added and the mixture is warmed with repeated shaking for about 2 min at about 60°C in a water bath. The mixture turns a light red-orange in the presence of alcohols.

Reagent:

1. Vanadate-quinolinol: 1.25g of 8-hydroxyquinoline are dissolved in 50 mL of 6 percent acetic acid. One mL of this solution is mixed with 1 mL of a 0.1 percent aqueous solution of Na_3VO_4 and then extracted with 30 mL of toluene. The toluene (upper) layer containing the quinoline-vanadate reagent is separated from the aqueous phase and can be used for analysis for 1 to 3 hr.

Notes:

1. Some nonalcoholic compounds give a purplish-brown which is not easily distinguished from the color of a positive reaction. Only light red-orange with a definite red tint should be considered as positive.

2. SH^- groups may interfere by shifting the color to a more yellow tint.

3. Some alcohols containing basic nitrogen or carboxyl groups give a negative response.

A.4.5.2.3 2,4-Dinitrophenylhydrazine (DNPH) test for aldehydes and ketones (RGN's 5 and 19)--

Procedure:

A small amount of the test material is dissolved in ethanol, and 1 drop of this solution is added to 5 to 10 drops of 2,4-dinitrophenylhydrazine reagent in a porcelain spotplate. When aldehydes or ketones are present a thick yellow precipitate appears after 10 to 20 sec which is easily visible even though the reagent is yellow.

Reagent:

1. 2,4-dinitrophenylhydrazine: Saturated in 2N HCL.

Note:

1. It is important to observe the ratio of reagent to test solution; the precipitate forms best at excess reagent concentrations.

A.4.5.2.4 Schiff's test for aldehydes (RGN 5)--
Procedure:

One drop of the test material in ethanol is added to 2 to 3 drops of fuchsin-sulfurous acid reagent. A bright deep-violet indicates the presence of an aldehyde.

Reagent:

1. Fuchsin-sulfurous acid reagent: 0.1g of fuchsin (p-rosaniline · HCL), 1.8g of $NaHSO_3$, and 2 mL of HCL are diluted with water to 100 mL. The reagent should be almost colorless.

2. Ethanol

Note:

1. Some compounds produce various shades of pink with the reagent. Only a definite violet is to be considered a positive test.

A.4.5.2.5 Hydroxamate test for esters (RGN 13)--
Procedure:

A few drops or equivalent amount of solid is added to 0.5 mL of hydroxylamine thymolphthalein solution. Then 2N potassium hydroxide in methanol is added dropwise until the solution turns blue. After addition of 5 drops excess NaOH, the solution is heated to boiling in a water bath and 2N HCL is added until the blue disappears. Then 1 to 2 drops of a 10 percent ferric chloride solution are added. The solution turns purple-red in the presence of esters. If a brown precipitate is formed 2 to 3 additional drops of ferric chloride are added. The color will then change to purple if esters are present.

Reagents:

1. Hydroxylamine thymophthalein solution: 7g of hydroxylamine · HCL is dissolved in 100 mL of methanol and 0.02g of thymolphthalein are added.

2. 2N potassium hydroxide: 11.2g of KOH is dissolved in 100 mL of methanol.

3. 2N HCL: 16 mL of concentrated hydrochloric acid is diluted to 100 mL.

4. 10 percent ferric chloride: 10g of ferric chloride is dissolved in 100 mL of water.

A.4.5.2.6 Iodine test for ethers (RGN 14)--

Procedure:

Five drops of iodine-carbon/disulfide solution are placed in a spotplate and 1 drop of the liquid or dissolved test material is added. In the presence of ethers, the purple color of the reagent changes immediately to a pale yellow or completely colorless. A change of color from purple to dark yellow or brown is not considered a positive result.

Reagent:

1. A few crystals of iodine are dissolved in carbon disulfide. The reagent should be light violet.

Note:

1. A decolorization of the reagent is not to be considered positive if later in PS 5D mercaptans are found to be present.

A.4.5.2.7 Ferric chloride test for phenols (RGN 31)--

Procedure:

One to two drops of the test material dissolved in chloroform are added to 1 drop of $FeCl_3$/pyridine reagent in a spotplate. A strong red, purple, blue, or green color indicates the presence of phenols. Excess reagent should be avoided.

Reagents:

1. $FeCl_3$ reagent: 1g of $FeCl_3$ is dissolved in 100 mL of $CHCl_3$. Then, 8 mL of pyridine are added and the mixture is filtered.

2. Chloroform

Note:

1. Sulfide or sulfhydryl groups interfere by producing a purple or green color also. Nitrophenols give a negative test.

A.4.5.3 PS 5C -- Tests For Functional Groups Containing Nitrogen--

A.4.5.3.1 Diphenylcarbonhydrazine test for carbamates (RGN 9)--

Procedure:

One drop of test solution or equivalent amount of solid is treated with 1 drop of phenylhydrazine in a small test tube. The mixture is heated to 195°C in an oil bath for 5 min. After cooling, 5 drops of nickel sulfate solution and 10 drops of chloroform are added and the mixture is shaken. A positive response is indicated by a red color in the chloroform (lower) layer. The color is best observed by holding the tube to a light.

Reagents:

1. Nickel sulfate: 5g of nickel sulfate ($NiSO_4$) is dissolved in 50 mL of water and 50 mL of concentrated aqueous ammonia is added.
2. Phenylhydrazine: Obtained from Aldrich Chemical Company.

Notes:

1. Many organic compounds turn brown when they are heated to 195°C. Therefore, care must be taken to distinguish a red color from charring of an organic material.
2. Isocyanates and certain other organics give a positive test.
3. Chloroform

A.4.5.3.2 Glutaconic aldehyde test for hydrazines (RGN 8)--

Procedure:

One drop of test solution (or an equivalent amount of solid with 3 to 4 drops of ether) in a depression of a porcelain spotplate is treated (in order) with 1 drop of pyridylpyridinium chloride, 1 drop of 1N sodium hydroxide, and 1 drop of concentrated hydrochloric acid. A blood-red spot that appears a few seconds after the addition of the hydrochloric acid is a positive test (see note).

Reagents:

1. Pyridylpyridinium chloride: 1g of pyridylpyridinium chloride (Aldrich Chemical Company) is dissolved in 100 mL of water.
2. 1N sodium hydroxide: 4g of sodium hydroxide (NaOH) pellets are dissolved in 100 mL of water.
3. Hydrochloric acid: Concentrated (36 percent) hydrochloric acid (HCL) is used.
4. Ether

Note:

1. Some compounds, most notably substituted phenols, turn dark upon treatment with sodium hydroxide or hydrochloric acid. If a positive test is observed, refer to the results of solution/reactivity tests (PS 2) to determine whether the material darkens with acid or base. If the material darkens with acid or base alone, the test for hydrazine is not positive.

A.4.5.3.3 Dimethylaminobenzaldehyde (DMAB) test for azo compounds (RGN 8)--Procedure:

A drop of test solution or equivalent amount of solid in the depression of a porcelain spotplate is treated with a drop of 4 percent hydrochloric acid and a few granules of zinc and allowed to stand for 15 min. The resulting test solution is spotted onto filter paper and allowed to air dry (10 to 15 min). The paper is then spotted with a drop of p-dimethyl-aminobenzaldehyde. An orange to red spot indicates a positive test (see note).

Reagents:

1. 4 percent hydrochloric acid: 10 mL of 36 percent hydrochloric acid is diluted to 100 mL with water.
2. P-dimethylaminobenzaldehyde: Benzene is saturated with p-dimethyl-aminobenzaldehyde (Aldrich Chemical Company).
3. Zinc: Granular zinc (fine) is used.

Notes:

1. Azo compounds are intensely colored. If the test material is colorless, it does not contain an azo compound in appreciable quantity and the test need not be performed.
2. Certain other compounds give colored products with p-dimethylaminobenzaldehyde. To confirm a positive test response, the test should be repeated without the treatment with zinc and hydrochloric acid. A similar response to p-dimethylaminobenzaldehyde indicates that the material is not an azo compound.
3. A decolorization of the test material will normally occur during the reaction with zinc and hydrochloric acid if the compound is an azo compound.
4. Benzene is a carcinogen and must be handled with appropriate caution!

A.4.5.3.4 Oxamide test for amides and nitriles (RGN's 6 and 26)--
Procedure:

A small portion (2 to 3 drops or equivalent) of test material is mixed with approximately twice the bulk amount of oxalic acid ($H_2C_2O_4 \cdot 2H_2O$) in a 3-in. test tube. The tube is heated carefully in a gentle flame until melting of the oxalic acid and subsequent bubbling occurs. Gentle heating is continued for an additional 10 to 15 sec, and sublimed oxalic acid is removed from the upper portion of the test tube by heating the upper part of the tube directly. The tube is cooled, and a small portion (about the size of a pea) of thiobarbituric acid is mixed with the contents of the tube, using a glass stirring rod. The mixture is heated in an oil bath at 120° to 140°C for 5 to 10 min. A positive response is indicated by a dark orange or red color.

Reagents:

1. Oxalic acid: Obtained from Aldrich Chemical Company.
2. Thiobarbituric acid: Obtained from Tridon (Fluka) Chemical Company.

Notes:

1. Care must be taken not to heat the mixture too strongly in the initial heating step.
2. Thiobarbituric acid is light orange but is easily distinguished from the dark orange of a positive test.
3. Many organics turn brown when heated. Care must be taken not to confuse this with a positive test.
4. Concurrent running of a blank with oxalic acid is recommended.

A.4.5.3.5 Tetrabase fusion test for organic nitro compounds (RGN 27)--
Procedure:

A drop of test solution or equivalent portion of solid test material is treated with a drop of tetrabase solution in a 3-in. test tube. If the material does not dissolve in the reagent solution, the test material is dissolved in a few drops of ethanol before addition of the reagent. The tube is placed in a container of boiling water for 2 to 3 min, then withdrawn. A positive response is indicated by a yellow to orange color in the bottom of the tube.

Reagent:

1. Tetrabase solution: 5g of 4,4'-methylenebis (N,N-dimethylaniline) (tetrabase -- Tridon-Fluka Chemical Company) in 100 mL of toluene.

Note:

1. Care should be taken to ensure that water does not enter the test tube while heating in the water bath.

A.4.5.3.6 p-Nitrobenzene-diazonium Tetrafluoroborate Test for Phenols and Amines (RGN 31)--

Procedure:

A small amount of the test material is added to 3 to 4 drops of p-nitrobenzene-diazonium tetrafluoroborate reagent. A red, orange, or yellow precipitate indicates the presence of phenols or amines.

Reagent:

1. p-Nitrobenzene-diazonium tetrafluoroborate: 1g in 100 mL of water.

Note:

1. Ortho- and para-substituted phenols and amines give a negative reaction with the exception of picric acid and dipicrylamine which show a positive reaction since they form yellow to orange precipitates at the reaction pH. Interferences are: aromatic SH-groups and isocyanates that hydrolyze at acidic conditions to amines. p-Hydroxyacetophenone gives a negative reaction.

A.4.5.4 PS 5D -- Tests for Functional Groups Containing Sulfur--

A.4.5.4.1 Cupric chloride test for dithiocarbamates (RGN 12)--

Procedure:

To a mixture of 1 drop of the liquid test material or a small amount of the solid test material and 2 drops of methanol in a spotplate, 2 drops of cupric chloride acetic acid reagent are added. A brown precipitate or brown coloration indicates the presence of dithiocarbomates. When it is difficult to decide whether the precipitate or brown color constitutes a positive reaction, the addition of 1 to 2 drops of $CHCl_3$ will extract the brown product from the aqueous phase and even small amounts of dithiocarbomates can be observed.

Reagent:

1. Cupric chloride acetic acid: 1g of $CuCl_2$ is dissolved in 50 mL of water and 50 mL of acetic acid.

Notes:

1. The test can also be carried out in a test tube with larger amounts of test material and this is preferable when the addition of $CHCl_3$ is required for better color observation.

2. Copper nitrate ($Cu(NO_3)_2$) can be substituted for copper chloride for this test.

A.4.5.4.2 Lead acetate test for mercaptans (RGN 20)--
Procedure:

One drop of the dissolved test material is added to 1 to 2 mL of a saturated solution of lead acetate in ethanol. A lemon-yellow precipitate appears immediately when thiol groups are present.

Reagent:

1. Lead acetate: Saturated solution of lead acetate in ethanol.

Note:

1. The test can also be carried out in a spotplate with 0.5 to 1 mL of reagent and 1 drop of a more dilute solution of the test material.
2. Disulfide groups can be detected with this test after reducing the test material with 1 mL of a 1N solution of hydroxylamine · HCl in methanol and some zinc powder.
3. A white precipitate may be formed, but this should not be interpreted as a positive test.

A.4.6 PS 6 -- Inorganic Functionality Tests

A.4.6.1 Zirconium Alizarinate Test For Fluorides (RGN 15)--
Procedure:

Four drops of a mixture of equal amounts of zirconium nitrate and sodium alizarin sulfonate solution are placed in a spotplate and 1 to 2 drops of the test material dissolved in water or alcohol, or a small amount of solid are added. In the presence of fluoride ions the burgundy reagent turns yellow.

Reagents:

1. Zirconium nitrate solution: 0.05g of $Zr(NO_3)_2$ dissolved in 50 mL of water and 10 mL HCl.
2. Sodium alizarin sulfonate: 0.05g of sodium alizarin sulfonate in 50 mL of water.

Notes:

1. The test can also be carried out by placing a drop of the dissolved test material on a zirconium-alizarin test paper. The paper is prepared by soaking a filter paper first in a 5 percent solution of $Zr(NO_3)_2$ in 5 percent HCL and then in a 2 percent aqueous solution

of sodium alizarin sulfonate. The paper is then washed with water and dried.

2. Fluoride from organic fluoro compounds can be detected only after fusion with sodium.

A.4.6.2 Tests for Metals and Metal Compounds--

A.4.6.2.1 Phosphomolybdate test for metals (RGN 21, 22, 23)--
Procedure:

A small amount of test material is added to 2 drops of phosphomolybdic acid and placed on a spotplate. When elemental metals are present a deep-blue color develops around the test material forming a blue solution after 0.5 to 1 min and/or after stirring with a glass rod.

Reagent:

1. Saturated phosphomolybdic acid (Aldrich Chemical Company)

Note:

1. Aluminum did not give a positive reaction.

A.4.6.2.2 Sulfide test for metal ions (RGN 24)--
Procedure:

Two drops of the test solution, acidified with nitric acid (5 percent) are placed in a spotplate. After adding a few grains of zinc sulfide a black, grey, or yellow precipitate indicates the presence of metal ions. If the response is negative, ammonium sulfide is added until the solution turns slightly alkaline. A precipitate indicates the presence of heavy metal ions. Metallic elements or salts, which may be present in waste materials, must be dissolved (i.e., in nitric acid) prior to conducting this test.

Reagents:

1. Zinc sulfide powder (Chem-Service)

2. Nitric acid 5 percent

3. $(NH_4)_2S$ 30 percent in water (Chem-Service)

Note:

1. Strong oxidizing agents interfere by converting the sulfide ion to sulfur.

A.5 DEVICE FOR TEST MIXING HAZARDOUS WASTE MATERIALS

Two important criteria for evaluating the compatibility of two or more substances when mixed together are heat generation and gas evolution. Both can create hazards in the form of pressure increases and formation of toxic gases. They are measured with a simple mixing device which is easy to assemble and use under field conditions. A functional diagram of the device is shown in Figure A-9.

A.5.1 Description

The device consists of a 120-mL glass reaction vessel equipped with a rubber stopper with holes for a hand-operated glass stirring rod with a Teflon paddle, a thermocouple (K-type stainless steel, Omega), a buret for adding liquids, and Teflon tubing which is connected to a 50-mL impinger bottle. The temperature is measured with a battery-operated digital thermometer (Omega Model 727C). The reaction vessel and the impinger bottle are held in a wooden block; the cavity for the reaction vessel is deep enough to provide protection in the event of glass breakage but still permits visual observation of the reaction. The reaction vessel is held by a clamp and metal stand.

A.5.2 Cautions

Violent reactions during mixing can be avoided by mixing small amounts of the test materials in a test tube before carrying out the reaction in the mixing device. If the mixture creates heat or forms gases or fumes in test tube quantities no mixing with larger quantities is necessary. In case of unpredicted, high pressure buildup inside the mixing vessel, the release of the stopcock will prevent the glass vessel from shattering. The experiment should be carried out behind a safety shield to avoid accidents.

A.5.3 Procedure

The mixing tests are carried out by placing 3 to 5g of the solid or 3 to 5 mL of the liquid test material in the reaction vessel, then closing the stopper, and adjusting the thermocouple so it reaches into the test material. The impinger is filled with 10 mL of water and connected to the reaction vessel. The temperature of the test material is recorded before the second (liquid) test material is added from a buret, the stopcock is closed, and both materials are mixed by turning the stirring rod. The reaction is observed for foaming, color changes, fumes, and temperature changes. Gas evolution is observed as bubbles appearing in the impinger solution. If desired, the impinger solution can be analyzed for $S^=$, CN^-, $CO_3^=$, Cl^-, Br^-, I^-, SCN^-, and pH. The procedures outlined in Section A.4 and below are used, substituting the impinger solution in each case for the test solution. See Table A-5 for references.

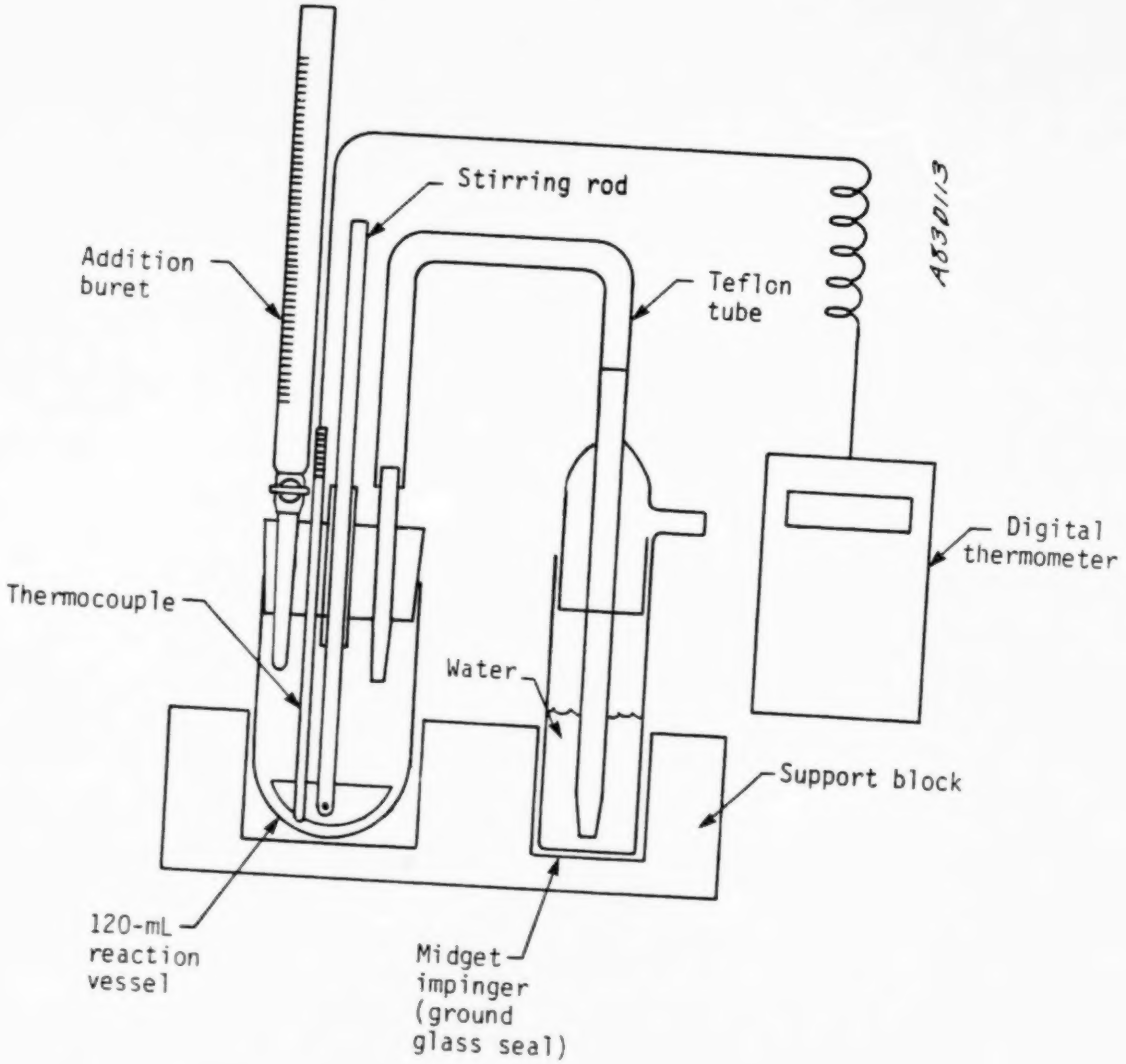

Stirring rod
Addition buret
Teflon tube
A830113
Digital thermometer
Thermocouple
Water
Support block
120-mL reaction vessel
Midget impinger (ground glass seal)

Figure A-9. Test mixing device.

TABLE A-5. REFERENCES FOR ADDITIONAL TESTS OF IMPINGER SOLUTIONS

Specie	Reference section
Sulfide ($S^=$)	A.4.4.1.2
Cyanide (CN^-)	A.4.4.1.1
Carbonate ($CO_3^=$)	A.5.4.1
Halide (Cl^-, Br^-, I^-)	A.4.4.1.3
Thiocyanate (SCN^-)	A.5.4.2

A.5.4 Additional Indicator Tests for Carbonate and Thiocyanate

The two following tests can be performed on the mixing device impinger solution to determine if carbon dioxide or thiocyanate has been evolved. Reagents for these tests are not, in general, in the kit. These tests do not identify RGN's but add information to the results of mixing wastes.

A.5.4.1 Barium Nitrate Test for Evolution of Carbon Dioxide--
Procedure:

Approximately 1 mL of the mixing device impinger solution is treated with 2 to 3 drops of 0.1N barium nitrate solution in a 3-in. test tube. If the solution turns cloudy or a white precipitate forms then carbonate is presumed to be present. Compare with a blank (unused) portion of impinger solution. The presence of carbonate indicates that carbon dioxide has been evolved.

Reagent:

1. Barium/nitrate solution: 2.6g of barium nitrate are dissolved in 100 mL of water.

A.5.4.2 Ferric Chloride Test for Thiocyanate--
Procedure:

Test the impinger solution for pH. Half fill a 3-in. test tube with the solution. If the solution is acidic, adjust the pH to slightly basic with 1 to 2 drops sodium hydroxide solution. Add approximately 5 drops of 10M sodium carbonate. Warm gently, if necessary, to dissolve. Acidify the solution with 6M hydrochloric acid. Add 1 to 2 drops ferric chloride solution. A blood red solution is a positive test for thiocyanate.

Reagent:

1. Saturated sodium carbonate solution: Add sodium carbonate with stirring to warm water until no more dissolves. Decant solution into storage container.

2. Ferric chloride: 1.35g of ferric chloride ($FeCl_3$) is dissolved in 100 mL of distilled water and 2 mL of concentrated hydrochloric acid.

A.5.5 Equipment Maintenance and Reagent Refill

A.5.5.1 Equipment Cleaning--

Test tubes, spatulas, spotplates, and watchglasses are cleaned between tests with acetone, methanol, or detergent (Alconox) and water. For this purpose, 2,000 mL acetone, 2,000 mL methanol, about 100g of Alconox, test tube brushes, and cleaning tissue (Kimwipes®) are included in the test kit. If no deionized water is available at the test site it can be either carried in the test kit or a deionizing cartridge (Barnstead D8950) can be attached with plastic tubing to a tapwater outlet. Deionized water is sometimes available at local supermarkets. Sufficient quantities of test tubes were carried during field testing so that used ones needed to be rinsed only at the site. Final cleaning could be carried out in a laboratory after return from the test site. Disposable Pasteur pipets are used for sample transfer and spot testing.

Depending on the solubility of the materials used in the mixing test, the reaction vessel, stirring rod, addition buret, and thermocouple are cleaned with acetone or methanol. They are then rinsed with deionized water and wiped dry with cleaning tissue. The impinger and the connecting tube are rinsed with deionized water. After completion of the mixing tests all equipment belonging to the mixing device is cleaned and returned to its case.

A.5.5.2 Reagent Refill--

Several of the reagents used in the kit are stable for only a few weeks and need to be prepared fresh before each field trip. These are the pyridine-barbituric acid and iodine-carbon disulfide reagents. It is also necessary that all reagent bottles be at least half full, which is sufficient for about 100 specific tests. Some of the reagents have to be prepared fresh for each test (vanadate-quinolinol, potassium permanganate, acetone), their components are included separately in the reagent cases and are mixed according to the instructions given in the respective sections of the instruction manual. Reagents which degrade in less than 3 weeks are discarded and prepared fresh. These reagents and their observed degradation characteristics are:

- Fuchsin-sulfurous acid -- fading of color or color change to brown

- Ferric chloride-pyridine -- precipitation

- P-dimethylaminobenzaldehyde -- appearance of crystals
- Tetrabase -- appearance of crystals or darkening of color
- P-nitrobenzene-diazonium tetrafluoroborate -- large amount of precipitation
- Zirconium alizarinate -- fading or darkening of color

In general it is advisable to prepare fresh reagents if color changes or precipitates are observed and not to use reagents 3 to 4 months after preparation. All reagents are easily prepared according to the instructions given in the test manual.

ATTACHMENT A

REAGENTS FOR HAZARDOUS WASTE FIELD TEST KIT

Reagents	Quantity	Section[a]
Peroxide test strips	1 vial	(4.5.2.1)
pH paper	2 vials	
Methylene blue paper	2 vials	(4.1.2)
2,6-dichloroindophenol paper	2 vials	(4.1.2)
KI-starch paper	2 vials	
Lead acetate paper	2 vials	
Sulfuric acid (concentrated)	50 mL	
Hydrochloric acid (concentrated)	50 mL	
Nitric acid (concentrated)	50 mL	
Sodium hydroxide (3M)	50 mL	
Hexane	100 mL	
Toluene	100 mL	
Methanol	100 mL	
Acetone	250 mL	
Hydrochloric acid (3M)	100 mL	
Nitric acid (3M)	100 mL	
Sodium pellets	50g	
Chloramine T reagent	25 mL	(4.2.2)
Pyridinebarbituric acid	25 mL	(4.2.2)
Ferric chloride	25 mL	(4.4.1.1)
Sodium pellets	100g	
Silver nitrate solution (0.1M)	25 mL	(4.4.1.3)
Ammonium molybdate -- HCl	25 mL	(4.4.1.4)
Stannous chloride -- glycerol reagent	25 mL	(4.4.1.5)
Sodium vanadate solution	50 mL	(4.5.2.2)
Quinolinol/acetic acid	50 mL	(4.5.2.2)
Toluene	250 mL	
Ethanol	150 mL	
Dinitrophenylhydrazine reagent	25 mL	(4.5.2.3)
Fuchsin/sulfurous acid reagent	25 mL	(4.5.2.4)
Hydroxylamine-thymolphthalein reagent	25 mL	(4.5.2.5)
Potassium hydroxide/methanol	25 mL	(4.5.2.5)
Ferric chloride (10%)	25 mL	(4.5.2.5)

[a] Indicates section in which directions for preparation are given

Reagents	Quantity	Section[a]
Iodine/CS_2	25 mL	(4.5.2.6)
Ferrox reagent	25 mL	(4.4.2)
Ferric chloride/pyridine reagent		(4.5.2.7)
Phenylhydrazine	25 mL	(4.5.3.1)
$NiSO_4$ reagent	25 mL	(4.5.3.1)
Chloroform	150 mL	
Pyridylpyridinium chloride	25 mL	(4.5.3.2)
Sodium hydroxide (1M)	25 mL	(4.5.3.2)
Hydrochloric acid (4 percent)	25 mL	(4.5.3.3)
Dimethylaminobenzaldehyde solution	25 mL	(4.5.3.3)
Granulated zinc	25g	
Oxalic acid (solid)	50g	
Thiobarbituric acid (solid)	50g	
Tetrabase solution	50 mL	(4.5.3.5)
Cupric chloride-acetic acid solution	25 mL	(4.5.4.1)
Lead acetate-ethanol reagent	25 mL	(4.5.4.2)
Hydroxylamine hydrochloride (1M)	25 mL	(4.5.4.2)
Zirconium-alizarin test paper	1 vial	(4.6.1)
Potassium permanganate/acetone	50 mL	(4.5.1.1)
Aluminum chloride	50g	(4.5.1.2)
p-Nitrobenzenediazonium tetrafluoroborate	50 mL	(4.5.2.8)
Phosphomolybdic acid	50 mL	(4.6.2.1)
Zinc sulfide	25g	
Ammonium sulfide	25 mL	(4.6.2.2)

[a] Indicates section in which directions for preparation are given

ATTACHMENT B

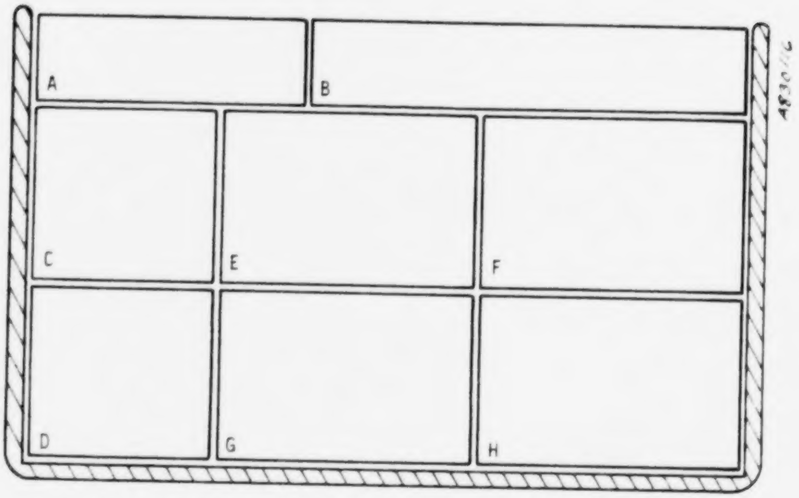
EQUIPMENT FOR HAZARDOUS WASTE FIELD TEST KIT

Equipment	Quantity	Cooler	Box
Porcelain spotplates	6 each	2	B
Spatulas (stainless steel)	3 each	1	A
Ceramic spoon	3 each	1	A
Pasteur pipets	2 boxes	2	A
Wire loops	3 each	1	A
Glass rods	15 each	1	A
Beakers (250 mL)	3 each	2	D
Beakers (150 mL)	6 each	2	D
Lighter for propane torch	2 each	1	B
Propane torch	1 each	1	B
Extra flame tips	3 each	1	B
Extra propane cylinders	1 each	3	K
Forceps	2 each	1	C
Test tubes (4 in.)	2 boxes	3	H
Test tubes (3 in.)	2 boxes	3	I
Test tube racks	2 each	1	C,D
Test tube holder	4 each	1	A
Funnels	10 each	2	C
Pipet bulb (large)	2 each	2	A
Pasteur pipet bulbs	12 each	2	A
Bottle with oil (glycerol)	1 each	3	L
Filter paper (4 in.)	2 boxes	2	B
Kimwipes	2 boxes	2	D
Large wipes	1 box	2	D
Watchglasses	6 each	2	B
Separatory funnels (30/60/125 mL)	1 each	2	D
Graduated pipets (5 mL)	2 each	2	D
Graduated pipet (10 mL)	1 each	2	D
Scissors	1 each	1	B
Screwdriver	1 each	1	B
Pliers	1 each	1	B
Parafilm	1 roll	2	B
Sample vials	30 each	3	G
Rack for vials	1 each	3	G
Clamps	2 each	2	D

Equipment	Quantity	Cooler	Box
Ring support	1 each	2	D
Stand	1 each	2	D
Wire screens	3 each	1	B
Acetone	2,000 mL	3	C
Methanol	2,000 mL	3	C
Glycerol	250 mL	3	L
Safety equipment			
Tyvac coveralls	3 each	3	
Safety shield	1 each	2	
Latex gloves	1 box	3	A
Solvent gloves	1 pair	3	D
Safety glasses	2 pairs	3	E
Safety goggles	2 pairs	3	E
Respirator	1 each	3	E
Waste solvent container	1 each		
Garbage bag(s) for disposal of contaminated pipets, etc.	As needed		
Mixing device		3	B
Glass vessels	2		
Buret	2		
Stopper no. 10 with 4 holes	2 or 3		
Tygon/Teflon tubing (approximately 8 in. long)	2 pieces		
Impinger bottle	2		
Glass stirring rod/paddle	1		
Thermocouple, digital readout	1		
Support/wooden rack	1		
Miscellaneous items			
Pencils, pens, markers			
Labels, forms			

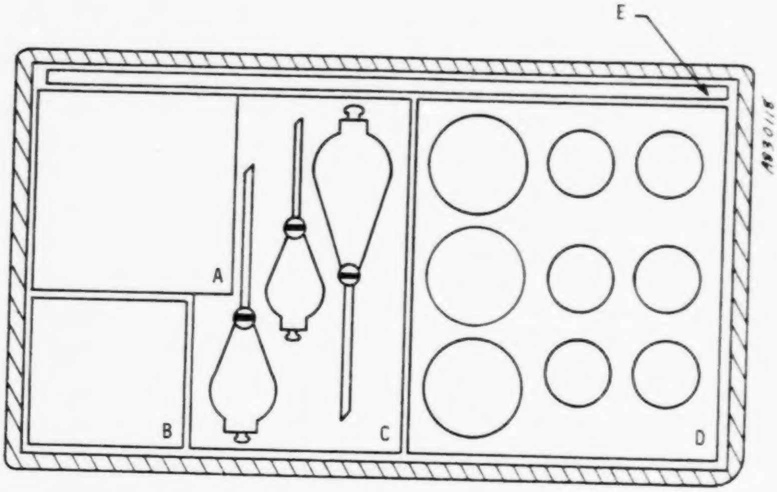
ATTACHMENT C

KIT ORGANIZATION



A	Spatulas, ceramic spoons, wire loops, glass rods, test tube holders, pipet bulbs
B	Lighter, flame tips, stand for propane torch, forceps, wire screens, scissors, pliers, screwdriver
C	Test tube rack and 4-in. test tubes
D	Test tube rack and 3-in. test tubes
E	Reagents for PS 1, PS 2, PS 6
F	Reagents for PS 4, PS 5A
G	Reagents for PS 5B
H	Reagents for PS 5C, PS 5D

Equipment organization -- cooler 1.



A Pasteur pipets

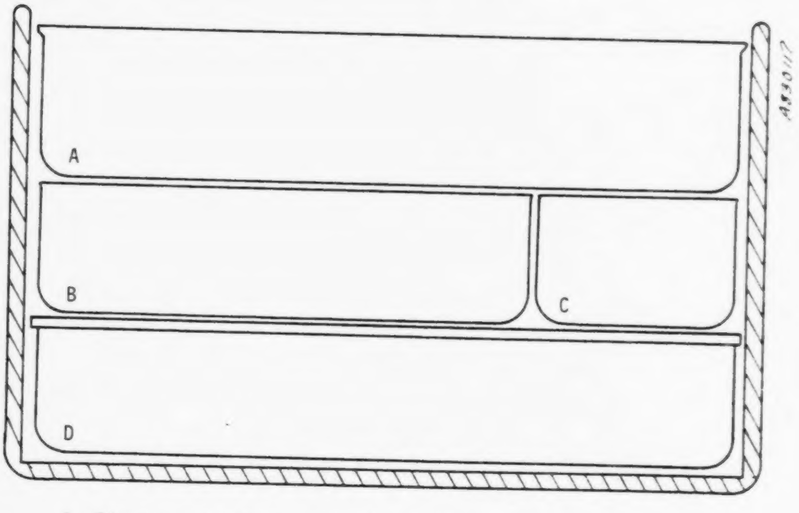
B Spotplates

C Separatory funnels

D Beakers

E Safety shield

Equipment organization -- cooler 2.



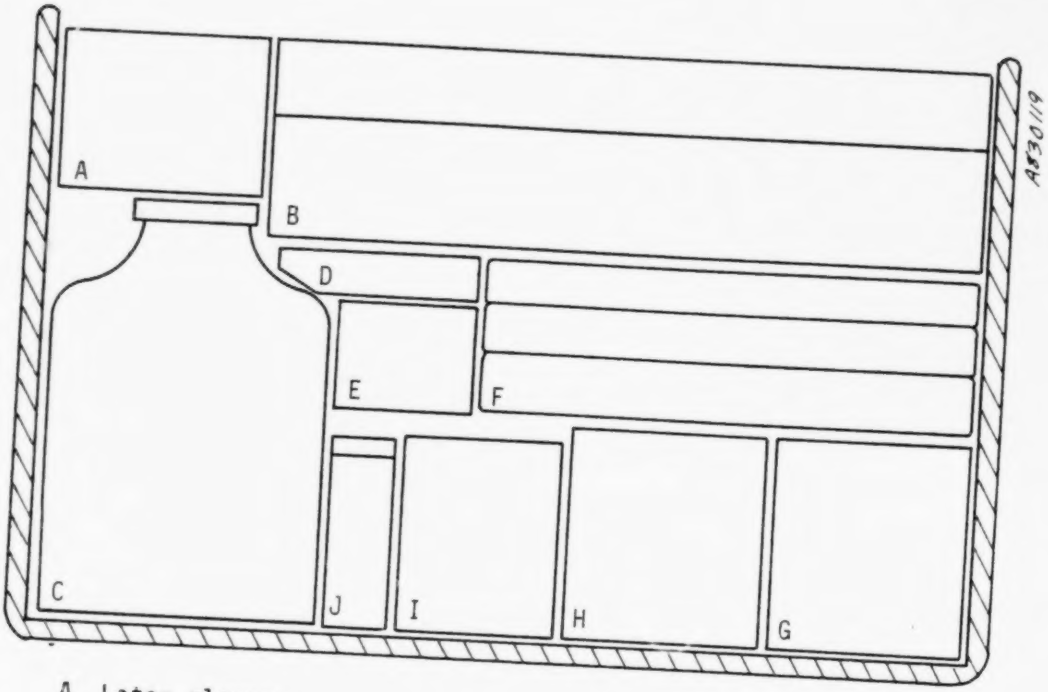
A Pipets, spotplates, funnels, beakers

B Filter paper, watchglasses, parafilm, extra sample vials

C Pencils, pens, markers, labels, tape

D Clamp, stand, ring support, kemwipes, paper towels, pipets

Equipment organization -- cooler 2 (top tray).



A Latex gloves

B Mixing device

C Wash solvents (acetone, methanol)

D Acid gloves

E Respirators, goggles

F Safety suits

G Rack with sample vials

H Test tubes, 4 in.

I Test tubes, 3 in.

J Sodium hydroxide and barium nitrate solutions

K Propane tanks (2) } Behind cutaway

L Glycerol }

Equipment organization -- cooler 3.